EMERSON ELECTRIC OF ST. LOUIS

ELECTRONICS AND SPACE DIVISION



Report Number 1675

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PERFORMANCE EVALUATION OF "THERMO-LAG" MATERIAL FOR ENTRY HEAT PROTECTION OF ADVANCED MANNED SPACECRAFT

FINAL REPORT October 1, 1962 - January 31, 1964

Volume Four - Blister Investigations

Prepared For National Aeronautics and Space Administration Manned Spacecraft Center Houston 1, Texas

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ABSTRACT

of sprayed "THERMO-LAG" T-500 material along the sidewalls of hemispherical cone models tested in an air-arc plasmajet on 10 December 1962. The preliminary investigation consisted of a chemical analysis of blistered specimens showing a nonhomogeneous concentration of salt. Since several components of "THERMO-LAG" T-500 have a sedimentation tendency, improper spraying techniques could have caused layers more concentrated in salt to be covered by layers more concentrated in resin. Therefore, the spray techniques were investigated as a possible cause of blistering.

Air-arc plasmajet tests of prepared models indicated that the spray technique was the cause for blistering of the material. The models were tested under the same simulated conditions as those that blistered in the air-arc plasmajet tests conducted 10 December 1962. The models specifically prepared to blister produced the identical type of blisters found in the original defective models. Photomicroscopic examination of both models showed that uniform distribution of the salt in the properly prepared, nonblistered model was very clearly distinguishable from the high concentration of salt in the blistered model.

As a result of this study, Emerson Electric recommends the following corrective measures (now being adapted) to eliminate the cause of blistering:

- 1. Use of recirculatory spray equipment for application of material.
- 2. Application of photomicrographs as a material control measure.

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author



SECTION I

"THERMO-LAG" T-500 is being investigated and characterized for NASA for application to the typical aft body of a super circular re-entry type vehicle under Contract NAS 9-877. An objective of the work being conducted under the contract is to perform a series of material simulation tests for the magnitude of aft body heating comparable to conditions typical of the vehicle.

The utility of "THERMO-LAG" T-500 as a subliming thermal parrier material has been illustrated, by numerous plasmajet tunnel tests. The physical composition of plasmajet tunnel post-tested "THERMO-LAG" T-500 material is qualitatively presented in the following test reports:

- 1. Emerson Electric Report 1414, "A Comparison of Thin Coatings of Phenolic Nylon and "THERMO-LAG" T-500 During Exposure to Low Convective Heat Fluxes of Long Duration."
- 2. Emerson Electric Report 1139, "Properties of "THERMO-LAG" T-500 EX167 Subliming Compounds."

Models tested for Emerson Electric Report 1414 were subjected to a heat flux of 30 BTU/FT² - SEC at an enthalpy of 3500 BTU/LB. The test conditions for Emerson Electric Report 1139 were approximately 596 BTU/FT² - SEC and an enthalpy of 16,000 BTU/LB. Inspection of the models indicated that the "THERMO-LAG" material maintained original stagnation and sidewall model configurations with formation of a nonreceding debris layer. Microscopic examination of those models tested for Emerson Electric Report 1414 revealed that most of the salt had sublimed away, providing proper material performance.

A series of air-arc plasmajet tests of hemispherical cone models tested for sidewall heating conducted under the test program of Contract NAS 9-877 on 10 December 1962 showed material blisters along the sidewalls of the models. To resolve the cause of blistering of sprayed material, a task analysis was conducted on the formulation and process of the tested "THERMO-LAG" T-500 material. The results of the test and the task analysis with post-test data to prove out the cause of the material blistering are presented in this report. The Quality Control Sheets of the GO - NO GO blistered models are presented in Appendix C.



SECTION II OBJECTIVE OF PROGRAM

The objectives of this program are:

- 1. To conduct a chemical and physical analysis to determine the cause of blistering of the material in the air-arc plasmajet tests on 10 December 1962.
- 2. To conduct necessary air-arc plasmajet tests to analyze the cause of blistering and establish a corrective measure.
- 3. To establish a procedure for use of the corrective measure in quality control of the material.

The essential purpose of the investigation of material blistering will be to determine whether the chemical composition of the material, its applicational technique, or their combined effect was the cause of the material failure. The approaches to examining the cause of blistering that consider the complete process of the material will be conducted along the following manner:

- 1. Hypothesize the phenomena to the specified material formulation, and determine by chemical analysis whether that composition possesses blistering characteristics.
- 2. Determine if the allowable application of the material for the blistered models contributes to the material failure.



SECTION III PRESENTATION OF THE PROBLEM

PERFORMANCE OF BLISTERED "THERMO-LAG" T-500 MODELS.

TEST TUNNEL CONDITIONS.

In accordance to the contract requirements specified in Section 1.4.1, Exhibit "A", sidewall heat tests were instituted on 10 December 1962. The tests were conducted in the hyperthermal plasmajet facility of Plasmadyne Corporation, Santa Ana, California. The 300KW are plasma system is described in detail in Emerson Electric Report 1414.

The tunnel performance data that would simulate the required magnitude of heating is tabulated in Table I.

TEST MODELS.

The models tested were hemispherical cones. The hemispherical nose bodies were made by pressure molding "THERMO-LAG" T-500 molding powder into blocks and machining these blocks to the desired configuration. The sidewalls of the steel cones, of 0.030-inch thickness, were built-up with "THERMO-LAG" T-500 by repeated spray applications and then machined to the desired material thicknesses.

Four of the nine models tested had nose radii of one inch, and the others had 5/8-inch nose radii. The thermocouples for measuring sidewall heating were positioned three inches behind the nose along the x-axis at 90° quadrants. The physical descriptions of the models tested have been tabulated along with the tunnel test conditions and are shown in Table I.

TEST RESULTS.

Five test models were chosen that summarize the results of the models tested. Photographs of the side views of these models tested (M-9, M-10, M-16, M-18, and M-22) are given respectively by Figures 3-1, 3-2, 3-3, 3-4, and 3-5. The photographs, approximately 1.55 size enlargements, illustrate blistering in the form of numerous small clusters to the occurrence of single large areas of blisters.

Cross-sections of models M-16, M-18 and M-22 were subjected to microscopic examination. The blister raised surfaces were verified to be caused by cohesive failure in the binder and did not proceed through to the substrate surface. A chemical analysis of models M-16, M-18, and M-22 was made and is discussed in Section IV.

The substrate surface temperature-time plots of Models M-9, M-10, and M-16 are plotted respectively in Figures B-1, B-2 and B-3 in Appendix B. The temperature-time plots of Models M-10 and M-16, being of longer duration, permit a better analysis of the effect of blistering on heat transfer. The temperature rise at the end of test runs are due to the salt having sublimed and leaving only the decomposing char layer as the thermal protector.

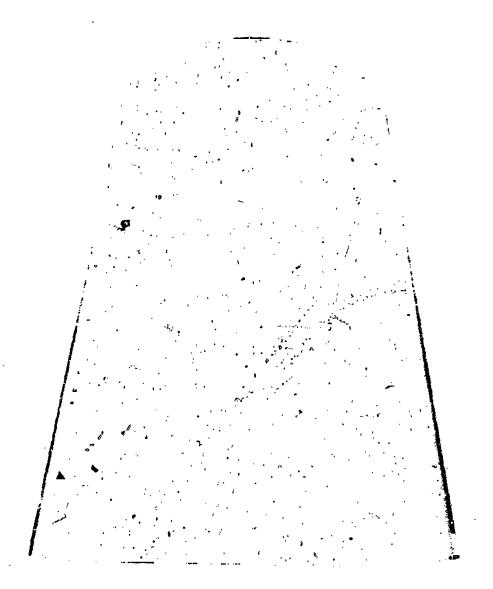
No corresponding test data is available to determine what deleterious effect, if any, blistering has upon the effectiveness of the material performance. Analytically, the increased voids in the material due to blistering would reduce the material thermal conductivity, but it would reduce also the material density.

Comparison of Figures 3-1, 3-3, 3-4, and 3-5 with Table I indicates that these photographs are the side views of the models that show the thin coating of material, while Figure 3-2 shows the side view for the thick coating of material.

Model Number	Stagnation Enthalpy .JTU/LB)	Moα 1 Stagnation Cold-Wall Heat Flux (BTU/FT ² - SEC)	Model Stagnation Pressure (ATM)	Tunnel Mass Flow (LB/SEC)	Model Radius (Inches)	Run Duration (SEC)	Overall Change in Weight (GM)	Overall Change in Length (Inches)	Sidewall Thermocouple	Number and Sidewall	Thickness (Inches)	4
M-6	14,890	110	. 00267	. 000546	1	30	8.3	. 039	. 038	. 034	. 100	. 100
M-9	9,865	110	. 0064	. 001448	1	150	25. 2	. 141	. 033	. 032	. 095	. 100
M-10	9,900	116	. 0064	.001448	1	300	47.3	. 346	. 036	. 034	. 093	. 099
M-18	10,100	215	. 016	. 00358	5/8	420	55.8	1. 112	. 051	. 049	. 099	. 100
M-16	17,365	170	. 005	. 006871	1	420	56.3	. 461	. 042	. 041	. 106	. 100
M-19	11,980	300	. 0 23	. 00491	5/8	300	51.0	. 902	. 050	. 051	. 100	. 100
M-21	17,658	300	.01	. 001748	5/8	45	10.9	. 078	. 050	. 052	. 097	. 092
M-22	17,680	300	.01	. 001748	5/9	90	16.0	. 169	. 053	. 051	. 100	. 100
M-23	17,500	300	.01	.001748	5/8	150	24.5	. 259	. 049	. 051	. 100	. 100
NOTE: A	Ul tests at 2	1% oxy	gen.									

Table I. Model Sidewall Data and Test Conditions





MODEL 9 TEST POINT 12

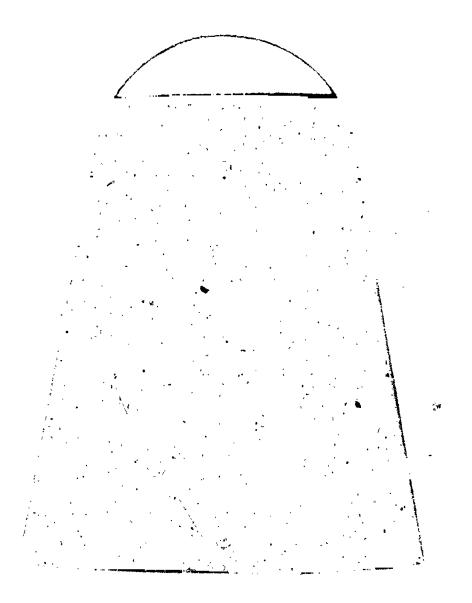
Figure 3-1. Side View of Test Model M-9



MODEL 10 TEST POINT 12

Figure 3-2. Side View of Test Model M-10





MODEL 16 TEST POINT 24

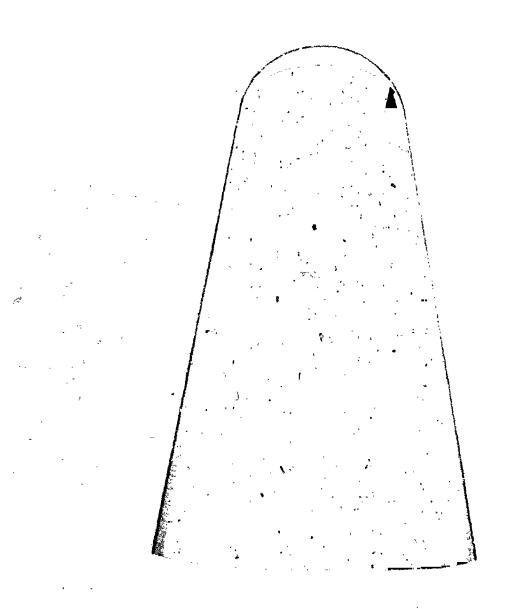
Figure 3-3. Side View of Test Model M-16



MODEL 18 TEST POINT 13

Figure 3-4. Side View of Test Model M-18





MODEL 22 TEST POINT 25

Figure 3-5. Side View of Test Model M-22



SECTION IV CHEMICAL ANALYSIS OF BLISTERED MODELS

PROCEDURE FOR CHEMICAL ANALYSIS.

To obtain positive assurance that the material in blistered models was properly compounded, chemical analysis of three models that blistered, M-16, M-18 and M-22, as well as samples of the original material were analyzed.

Samples of the char layer at the nose and at the side where blistering occurred were obtained as well as samples of the material beneath the char. To obtain a chemical analysis, the following analytical scheme was followed:

- 1. Dry sample at 60°C and 30-inch vacuum in a vacuum oven to determine loss of weight on drying.
- 2. Re-equilibrate to room temperature conditions to determine atmospheric moisture pick-up.
- 3. Extract three times to separate the NH₄BF₄ salt, then evaporate and weigh the salt.
- 4. Dry residue from above extraction and weigh. Ignite the dried residues at 1000°C and weigh. The difference between the dried and ignited residues is the total organic content of the material.

Attempts to separate the resin, unpolymerized polymer tricresyl phosphate and carbon black, all of which were included in the term total organic, were not quantitatively successful.

These analyses were performed on the three models and a sample of the original "THERMO-LAG" T-500 material which was used as a control.

RESULTS AND DISCUSSION.

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Results of the analysis are tabulated in Table II. The samples tested for M-18 were taken from sides numbered (1) and (2), in Table A, as were the samples from other models, unless indicated. Table II indicates that very little, if any, solvent was entrained in the "THERMO-LAG", ruling this out as a cause for blistering.

The close agreement of the analysis between the original T-500 and the virgin material of

the blistered samples of M-16 and M-22 are assurance that these samples were properly compounded.

Some differences in the composition of the clistered material for the thin coated sides is apparent. Model M-22 had a significantly higher NTABF4 content. The higher value is due in part to the fact that the model was subjected to a short exposure time of 90 seconds while the others were tested for 420 seconds. (See Figures 3-3, 3-4, and 3-5.) More significant, however, is the fact that the sample from model M-22 was taken from the bottom layers of material directly below a blistered spot. The samples from the other models were taken from material layers close to the blistered surface.

To summarize, the principal points of the findings of the chemical analysis of the virgin material beneath the blisters and the T-500 control material indicates that the composition of blistered and control materials are identical. With the chemical analysis showing that the salt concentration was greater in layers of material furthest from a blistered surface, the process whereby "THERMO-LAG" is applied, was then investigated as a cause for blistering.

		Mode	M-16	Mode	M-18	Model M-22		
	Original T-500	Charred Nose	Virgin Beneath	Blistered Side	Blistered Side	Blistered Side	Virgin Beneath	
% loss in weight on heating		1,59	0.20	1.47				
% gain in weight on standing 3 days		1.48	0.19	1.22				
NH ₄ BF ₄ , (%)	49.2	2.4	51.7	6.1	4.1	15.9	49.7	
Total organics, (%)	41.9	75.3	40.0	66.2	69.1	67.4	44.9	
SiO ₂ , (%)	8.9	22.3	8.3	27.7	26.8	16.7	5.4	
Ratio NH ₄ BF ₄ /total organics	1.17	0.03	1.29	0.09	0.06	0.24	1.11	

Table II. Chemical Analysis of Blistered Models



SECTION V MATERIAL APPLICATION TECHNIQUES

INVESTIGATIVE SPRAY TECHNIQUE.

SPRAY EQUIPMENT.

"THERMO-LAG" applied by spraying is suspended in a volatile solvent. Since several of the components of "THERMO-LAG" have a sedimentation tendency, the application by spraying requires meticulous attention to spray techniques to obtain a homogeneous coat that will possess the required performance characteristics.

In the preparation of the test models of 10 December 1962, "THERMO-I.AG" material was applied by spraying with a De Vilbiss Spray Gun (No. P-JGA-502-69-D). The spray gun application technique uses a simple pressure cup assembly (De Vilbiss type No. PKB 519) in which the operator attempts to keep the solids in homogeneous suspension by frequently stirring the material in the cup with a spatula. To assure homogeneous suspension of the solids, a more exact technique is available. This technique involves fitting the pressure tank with an fir motor driven agitator, which employs a recirculating pump. The device pumps the material through a bypass loop back into the tank, thus allowing no settling of solids. This apparatus is shown in Figure 5-1.

RESULTS OF SPRAY TECHNIQUES.

The spray gun type of equipment which uses no special agitation has a tendency to separate the material into three layers. The bottom layer being rich in the more dense components, namely, ammonium fluoborate and refrasil. The middle layer contains a solution of the resin in the solvent, and floating on top are the very light phenolic microballoons. Even when the pot is shaken by hand during spraying, mixture separation is difficult to prevent, requiring meticulous attention to obtain the desired results. The result of this type of separation is a nonhomogeneous coating of the material in which layers more concentrated in salt are covered by layers more concentrated in the resin. Because the polymeric material ablates at a somewhat higher temperature than the sublimation temperature of the salt, the salt would vaporize underneath the polymer film to form blisters, since little action would exist to form voids that would allow passage of the formed gases

The problem associated with sprayed material separation is not encountered using the stirred tank facility. The recirculating pump provides a homogeneous coating, deposited in thin layers of approximately five mils, with each layer being allowed to air dry before the next application. Each coat should have a wet appearance immediately after deposition and then be allowed to dry to a non-tacky state. A drying time of 20 minutes is required

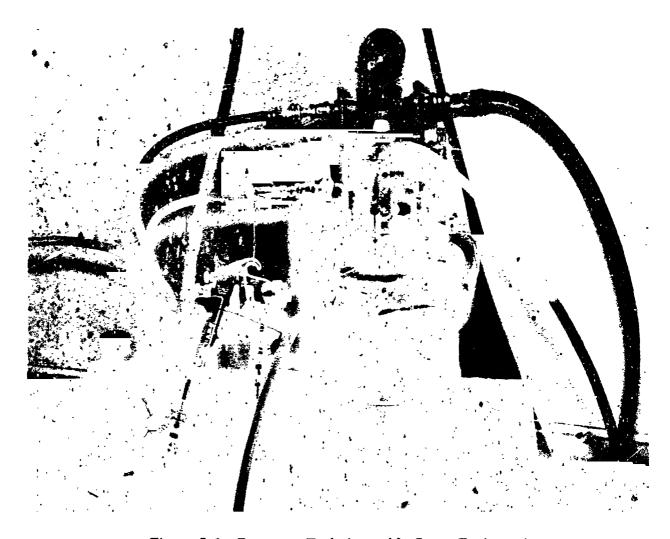


Figure 5-1. Pressure Tank Assembly Spray Equipment

at low humidity and at temperatures of 85-90°F. A lower temperature or high humidity necessitates a longer drying period. To interrupt the pattern of alternate application of layers and drying before the desired thickness has been obtained results in a longer drying time and a coat in which layers can be observed. The proper atmospheric conditions and surface preparation are essential to secure a consistent uniform and effectual application.

CURING CYCLE.

For proper utility of the "THERMO-LAG" material, the correct curing cycle is an essential process of quality control.

Curing of "THERMO-LAG" T-500 is accomplished by slowly raising the temperature from 110°F to 290°F while pressure is applied to the model. The pressure is applied



using a vacuum bag technique. The vacuum bag constructed of Mylar film is sealed at the edges with a sealing compound (Prestite Permagum 382). The evacuation of air from the bag causes it to collapse, which in turn, exerts pressure on the model and at the same time facilitates the removal of solvent and gaseous products. A vacuum of approximately 20-25 inches of water is applied and special precautions are taken to insure that the bag contains no leaks from pin holes in the mylar film or insufficient sealing of the edges. The curing is accomplished in the oven shown in Figure 5-2.

The curing cycle may be divided roughly into three temperature zones in which different chemical reactions occur. The temperature zone from 110-170°F is characterized by the slow removal of the low boiling solvents. The intermediate zone from 180° to approximately 220°F is distinguished by the removal of water, which is a by-product of the polymerization of the phenolic resin. The reaction, in which water is formed during the cross linking of the polymer chains, is described by the following generalized equation.

$$\begin{bmatrix}
-CH_2 - OH & CH_2OH \\
-CH_2 - OH & CH_2OH
\end{bmatrix}_n + \begin{bmatrix}
OH & CH_2 - \\
OH & CH_2 - \\
OH & CH_2 - \\
OH & OH
\end{bmatrix}_n$$

The higher temperature zone (250-290°F) is where the curing or cross linking of the acrylonitrile butadiene elastomer occurs. The reaction is catalyzed by zinc oxide and sulfides according to the following generalized equation.

$$\begin{bmatrix} -CN \\ -CH - CH_2 - CH_2 - CH = CH - CH_2 - \end{bmatrix}_n$$

$$\begin{bmatrix} -CH - CH_2 - CH_2 - CH - CH_2 -$$

Material that has been cured too rapidly will have soft and springy physical features due to solvent entrapment. These properties of a material can be detected readily by a hardness test and by an infrared heat test. The use of infrared heating causes the material to form blisters and bubbles, which distinguishes it from properly cured material which ablates in an even manner. The formation of such blisters differs considerably from the kind of blistering under consideration and would have been readily detected prior to plasmajet testing.

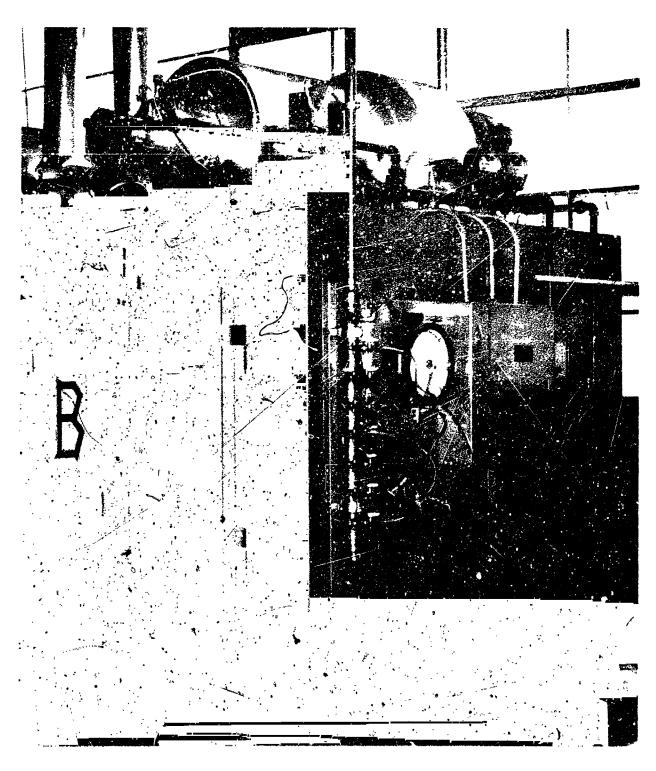


Figure 5-2. Curing Oven



SECTION VI GO - NO GO VERIFICATION PROGRAM

PREPARATION OF GO - NO GO BLISTER MODELS.

The second step in the objective of this program was to conduct air-arc plasmajet tests for verification of the analyzed cause of blistering of "THERMO-LAG" T-500 material.

MODEL DESIGNATION.

A chemical analysis of the blisters and blister areas of the models tested on 10 December 1962 showed both binder-rich, binder-poor, salt-rich, and salt-poor areas. Analysis of the test model series indicated that improper spraying and mixing of the material were the chief causes of blistering. Proof of this conclusion was to be established by a series of plasmajet tests.

To show that improper application of material was the primary cause of blistering, a series of models was constructed to simulate the previously observed conditions and produce the identical type of blisters found in the defective models. Three conditions based on the requirement by NASA that blisters be produced in specified areas and of specified size on particular models were employed for the test series. To ensure that each condition be met, arbitrary letter symbols were used to designate each series. In the D and E series a number was added to separate the thinner wall models from the thicker wall models for convenience during fabrication. Preparation of all tested models used quality control materials.

MODELS NOT EXPECTED TO BLISTER, GG SERIES.

"THERMO-LAG" T-500 is composed basically of a binder system, a salt, and sufficient solvent to make a sprayable composition. Unless agitated continuously, however, the coarser salt particles will rapidly drop out of suspension, collecting in the bottom of the spray container. The lighter resin components will stay in solution with the solvents. As discussed previously, material separation will contribute to defective models. The GG series of models was prepared to eliminate this cause. T-500 material from Lot B/N 10582 was mixed in a paint shaker assembly to completely disperse the naterial for spraying. The mixed material was applied by recirculating equipment. Models were built up in the normal manner until the desired thickness was achieved. The models were then air dried for the required time, placed in vacuum bags of mylar film, and cured in the manner specified for this program.

The test results for these models are presented in Table III. As illustrated in Figures A-1 through A-8 of Appendix A, no blisters occurred.

GG-2 10.179 109.5 75 18.47 .095 .034 .026 .027	Model Number*	Stagnation Enthalpy (BTU/LB)	Model Stagnation Cold-Wall Heat Flux (BTU/FT ² - SEC)	Run Duration (SEC)	Overall Chango In Weight (GM)	Overall Change in Length (IN.)	Sidewall	Thermocoupie Number and Sidewall Thickness	Comments	Average Surface Temperature (°R)	Time for Substrate To Reach 500°F	
GG-5 9,872 109 90 18.65 .123 .033 .025 .038	GG-2			75	18 47	095		<u></u>				
GG-8 9,987 126 35 8.15 048 .030 .030 .027	I	1]	}	ļ	Į	j	j	j			ĺ
GG-4 16, 101 109.5 150 30.15 .238 .108 .105 .104 3960 94.2 GG-7 9,865 109 780 106.68 1.246 .101 .107 .102 3960 99 GG-11 10,291 109.5 390 63.88 .582 .103 .102 .102 3930 101.9 E-15 9,801 109 75 16.41 .128 .044 .042 .041 .043 .043 E-16 10,024 109.5 75 15.87 .128 .042 .045 .043 .042 E-17 10,125 110 50 11.17 .061 .045 .043 .042 D-6 9,926 169.5 320 52.00 .436 .098 .105 .098 .3930 31.9 D-10 9,871 109 385 58.46 .531 .101 .104 .106 .3930 123.4 D-11 10,109 110 855 103.64 1.331 .101 .104 .106 .099 .3960 111.1 D-1 9,875 109 330 53.78 .505 .108 .106 .099 .3990 27.7 D-2-4 10,334 109.5 345 54.59 .540 .102 .108 .108 .3980 121.8	1	1	ĺ	1			1	1		d er		
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GG-4 16, 101 109.5 150 30.15 .238 .108 .105 .104 3960 94.2 GG-7 9,865 109 780 106.68 1.246 .101 .107 .102 3960 99 GG-11 10,291 109.5 390 63.88 .582 .103 .102 .102 3930 101.9 E-15 9,801 109 75 16.41 .128 .044 .042 .041 .043 .043 E-16 10,024 109.5 75 15.87 .128 .042 .045 .043 .042 E-17 10,125 110 50 11.17 .061 .045 .043 .042 D-6 9,926 169.5 320 52.00 .436 .098 .105 .098 .3930 31.9 D-10 9,871 109 385 58.46 .531 .101 .104 .106 .3930 123.4 D-11 10,109 110 855 103.64 1.331 .101 .104 .106 .099 .3960 111.1 D-1 9,875 109 330 53.78 .505 .108 .106 .099 .3990 27.7 D-2-4 10,334 109.5 345 54.59 .540 .102 .108 .108 .3980 121.8	į			1		i		1		pre		
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GG-11 10,291 109.5 390 63.88 .582 .103 .102 .102 3930 101.9 E-15 9,801 109 75 16.41 .128 .044 .042 .041 3930 31.9 E-16 10,024 109.5 75 15.87 .128 .042 .045 .043 .042 .3910 33.9 E-17 10,125 110 50 11.17 .061 .045 .043 .042 .041 .042 .044	GG-4					. 238] .108 [.105	.104	}	3960	94.2
E-15 9,801 109 75 16.41 .128 .044 .042 .041 3930 31.9 E-16 10,024 109.5 75 15.87 .128 .042 .045 .043 .042 E-17 10,125 110 50 11.17 .061 .045 .043 .042 E-18 10,035 109.5 50 15.20 .116 .041 .042 .041 .042 .041 D-6 9,926 169.5 320 52.00 .436 .098 .105 .098 D-10 9.871 109 385 58.46 .531 .101 .104 .106 D-11 10,109 110 855 103.64 1.331 .101 .104 .106 D-12 9,875 109 330 53.78 .505 .108 .106 .099 D-1 9,855 109 75 15.65 .113 .037 .033 .031 D-2 9,826 109 86 .6.75 .124 .033 .034 .035 D-2-4 10,334 109.5 345 54.59 .540 .102 .108 .108 3930 31.9 3930 31.9 3930 31.9 3930 31.9 3930 31.9 3930 31.9 3940 31.9 3950 31.9 3960 31.9 3960 31.9 3960 31.9 3960 31.9 3970 32.4 3970 32.4 3980 27.7 3980 121.8	GG-7	9,865	109	780	106.68	1.246	. 101	.107	. 102		3960	99
E-16	GG-11	10, 291	109.5	390	63.88	.582	.103	.102	. 102		3930	101.9
E-17 10,125 110 50 11.17 .061 .045 .043 .042 .041 .041 .042 .042 .0	E-15			75		. 1 28	ļ		. 041		3930	
E-18	E-16	10,024	109.5	75	15.87	.128	.042	.045	. 043		3910	33.9
D-E 9,926 169.5 320 52.00 .436 .098 .105 .098 .55 .098 .105 .098 .106	E-17	10, 125	110	50	11.17	.061	.045	.043	.042		3910	32.3
D-11 10,109 110 855 103.64 1.331 .101 .104 .106 3930 112.6 D-12 9,875 109 330 53.78 .505 .108 .106 .099 3960 111.1 D-1 9,855 109 75 15.65 .113 .037 .033 .031 3930 24.1 D-2 9,826 109 8C 16.75 .124 .033 .034 .035 3890 27.7 D-2-4 10,334 109.5 345 54.59 .540 .102 .108 .108 3980 121.8	E-18	10,035	109.5	50	15,20	.116	.041	. 042	.041	_च क्रि	3960	31.9
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D-11 10,109 110 855 103.64 1.331 .101 .104 .106 3930 112.6 D-12 9,875 109 330 53.78 .505 .108 .106 .099 3960 111.1 D-1 9,855 109 75 15.65 .113 .037 .033 .031 3930 24.1 D-2 9,826 109 8C 16.75 .124 .033 .034 .035 3890 27.7 D-2-4 10,334 109.5 345 54.59 .540 .102 .108 .108 3980 121.8	D-10	9.871	109	385	58.46	.531	. 101	.104	. 106	Bilis 8 pr	3930	123.4
D-1 9,855 109 75 15.65 .113 .037 .033 .031 3930 24.1 D-2 9,826 109 8C 16.75 .124 .033 .034 .035 3890 27.7 D-2-4 10,334 109.5 345 54.59 .540 .102 .108 .108 3980 121.8	D-11	10,109	110	855	103.64	1.331	. 101	.104	.106	,ē	3930	112.6
D-2 9,826 109 8C 16.75 .124 .033 .034 .035 3890 27.7 D-2-4 10,334 109.5 345 54.59 .540 .102 .108 .108 3980 121.8	D-12	9,875	109	330	53.78	. 505	.108	.106	.099		3960	111.1
D-2 9,826 109 8C 16.75 .124 .033 .034 .035 3890 27.7 D-2-4 10,334 109.5 345 54.59 .540 .102 .108 .108 3980 121.8	D-1	9,855	109	75	15.65	.113	.037	.033	.031		3930	24.1
D-2-4 10,334 109.5 345 54.59 .540 .102 .108 .108 3980 121.8	D-2		109	8 C	16.75	.124	.033	.034	. 035		3890	27.7
	D-2-4	'	109.5	345	54.59	.540	. 102	. 108	.108		3980	121.8
	D-2-5	10, 230	109.5	420	61.86	.676	.093	.104	.103		3940	25 3

*All models run at test point 12; stagnation pressure 0.0064 atmosphere; oxygen concentration 21 percent; tunnel mass flow .001448 LB/SEC.

Table III. Model Sidewall Data and Test Conditions



MODELS EXPECTED TO BLISTER, D SERIES.

The D series of tested models was prepared to predict blisters in certain areas. The material to be prepared for spray was taken from Lot B/N 10582 and mixed with appropriate solvent in the paint shaker. After mixing, the material was allowed to stand for ten minutes. Following the settling period, the material in the top half of each container was carefully poured into a clean container and arbitrarily labeled AA. The material remaining in the container was arbitrarily labeled XX.

The next procedure to prepare the material for spraying was the addition of material from Lot B/N 10662 to the XX material. The materials were mixed in the paint shaker, followed by a settling period of 15 minutes. The top two-thirds of the solution was then poured into a clean can labeled BB. The remaining one-third was labeled CC. Enough solvent was added to the CC material, to make it sprayable. The first spray application to the models was a prime coat of normal-mix "THERMO-LAG" sprayed on the substrate sleeves. Using a masking technique, 90° quadrants were then alternately sprayed with XX and CC material for four coats. The models were then sprayed with five coats of BB material. The final coating was a spray application from material Lot B/N 01223. The sprayed material was cured through normal procedure. Following curing, two models were machined to 31-to 37-mil wall thickness and arbitrarily coded D-1 and D-2. The other model was finished to 93- to 108-mil wall thickness and coded D-2-4.

Test results of the prepared material models are presented in Table III. Figures A-9 through A-11 in Appendix A illustrate the ability of the spray technique to produce large blisters in opposite 90° quadrants as specified.

MODELS EXPECTED TO BLISTER, E SERIES.

The E series of tested models was prepared per NASA request to predict blisters of specified size and location. The models were prepared by first spraying the substrate sleeves with a prime coat of standard "THERMO-LAG" material. Five coats of the AA material were applied to the prime coat. To produce the required blister sizes of approximately $1/2 \times 1/2$ inch square in the center of the sleeve body and 180° apart, a means was devised for the task. A mask was fabricated from a piece of cardboard with the required size hole. After each of the coats of the AA material was sprayed, the model was again sprayed locally using the mask in the designated location. The procedure was repeated with the previously prepared CC material. The entire models were then sprayed with three coats of AA material and a final coating of normal-mixture T-500. The vacuum bag and curing operations were conducted using normal procedures.

Models labeled D-9, D-10, D-11, and D-12 listed in Table III are also of E series models. These models were mislabeled D on the sleeves prior to spraying, and the designations were kept on the model proper.

Figures A-12 through A-19 in Appendix A illustrate that the models produced by the

outlined techniques did blister at the designated locations, with possible small blister spots randomly located on the rest of the model bodies.

MODEL PERFORMANCE.

TEST CONDITIONS.

The test conditions for the series were chosen by NASA and pointed out in a Memorandum of Understanding between the Emerson Electric Manufacturing Company and NASA. This memorandum stated that the test condition would be the previously calibrated test point 12. Nominal tunnel operating parameters, at this test point, were:

- 1. Stagnation enthalpy: 10,000 BTU/LB.
- 2. Model stagnation cold-wall heat flux: $110 \text{ BTU/FT}^2 \text{SEC}$.
- 3. Model stagnation pressure: 0.0064 atmosphere.
- 4. Nozzle mass flow: 0.001448 LB/SEC.
- 5. Oxygen concentration: 21 percent.

These test conditions were identical to those for blistered models M-9 and M-10 of the original test series of 10 December 1962. Close inspection of Tables I and III indicate that the actual test conditions did not vary from this nominal test point by more than three percent. Therefore, it may be stated that the conditions present in the original test series were accurately duplicated in the present series.

The models were tested in the Mach 3, 200KW, simulated air plasmajet of the Plasmadyne Corporation.

TEST MODELS.

The test models used for the blistered verification series were all nominally two inches in diameter. The model configuration was a hemisphere-cylinder with an over-all length of four inches. The forebody hemispherical portion was solid molded material. The aftbody cylindrical portion was material sprayed on a 0.02-inch thick steel substrate. Instrumentation of the cylindrical portion consisted of three 28-gage chromel-alumel thermocouples spot-welded to the inside of the steel cylinder 120° apart and one inch from the rear of the model. The two basic material thicknesses of 0.1 inch and 0.03 inch utilized in the original tests were duplicated here to insure accurate reproduction of the original test series.



TEST RESULTS.

A summary of test results is presented in Table III. An important factor that cannot be presented in tabular form is that the models predicted to blister did so after approximately 40 seconds. This was the approximate time that the models of the original series blistered at this test condition. Thereby, the duplication of results was achieved.

By comparing the time for equal-thickness sidewall models to attain 500°F, some comparison may be made between the thermodynamic effectiveness of material that blistered and material that did not blister. Table III presents this time data. From this table it would seem that blistering has no deleterious effect on the thermal shielding property of the material. The data, in fact, shows that the material that blistered was more effective as a heat shield. The substrate temperature-time plots of the Go - No Go tested models are presented in Appendix B (Figures B-4 through B-22).

The probable efficiency of the tested models expected to blister and those not expected to blister can be made from a study of Figures 6-1 and 6-2. These figures record the sidewall heat transfer test data envelope for the substrate temperatures and corresponding imposed heat loads for duration of model exposure in the plasma stream. The heat load-temperature envelope gives the entire range of data experienced for all models tested. An analysis of the heat flux envelope indicates that for a given duration of exposure in the plasma stream the nonblistered models presented better protective coating for their thermal environment.

The results of the sidewall heating tests for model sections having a nominal material thickness of 0.03-inch of "THERMO-LAG" T-500 are recorded in Figure 6-1. To illustrate the utility of the heat transfer envelope, representative calculated points for a typical blistered model, M-2-D, and a nonblistered model GG-5, are shown. From sidewall calorimeter data for the sidewall heat flux of 17 BTU/FT² - SEC, an exposure time of 42 seconds yields a total heat load of 714 BTU/FT². For the heat load, the temperatures occurring after 42 seconds were plotted on the graph as indicated.

A similar comparison is indicated in Figure 6-2 for models having a nominal material thickness of 0.1 inch of "THERMO-LAG" T-500. The representative models plotted are D-9 and GG-1A.

The models used for comparison represent nonblistered models (GG-1A and GG-5), models expected to produce large blisters in specific areas (C-2), and models prepared to produce many small blisters at specific locations on the sidewalls (C-9).

An explanation of the difference in effectiveness, for the exposed heating time, of the blistered and nonblistered material may be due partially to the lower thermal diffusivity of the blistered material caused by the formation of many voids beneath each blister.

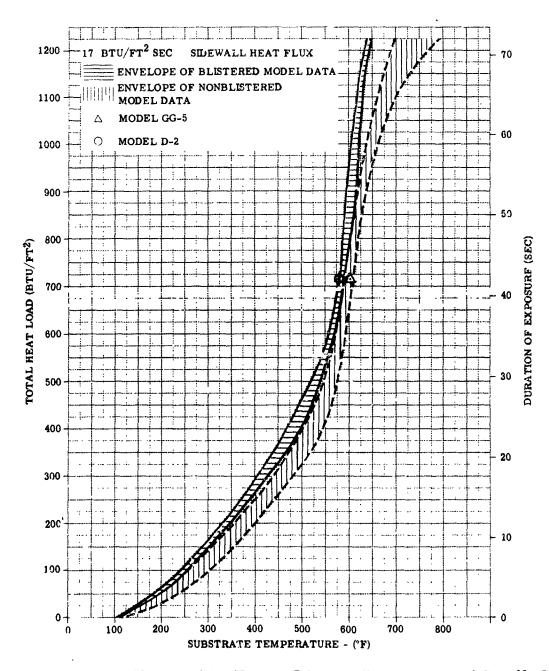


Figure 6-1. Total Heat Load for Various Substrate Temperatures of Go - No Go Blistered Model Tests (0.03-Inch Sidewall Thickness)



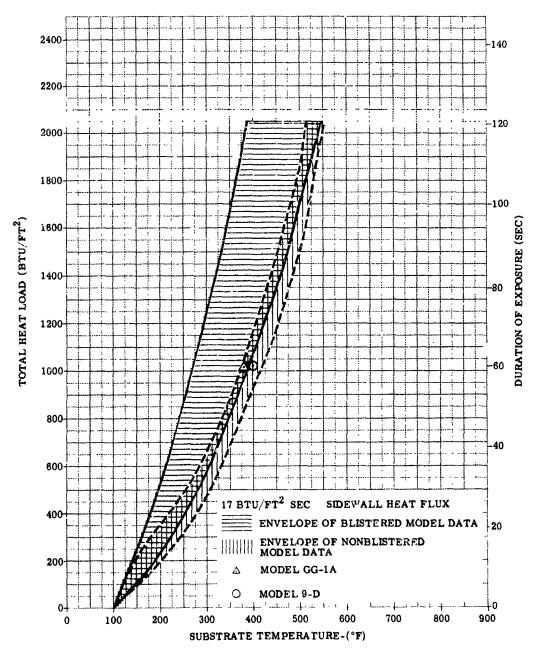


Figure 6-2. Total Heat Load for Various Substrate Temperatures of Go - No Go Blistered Model Tests (0.1-Inch Sidewall Thickness)

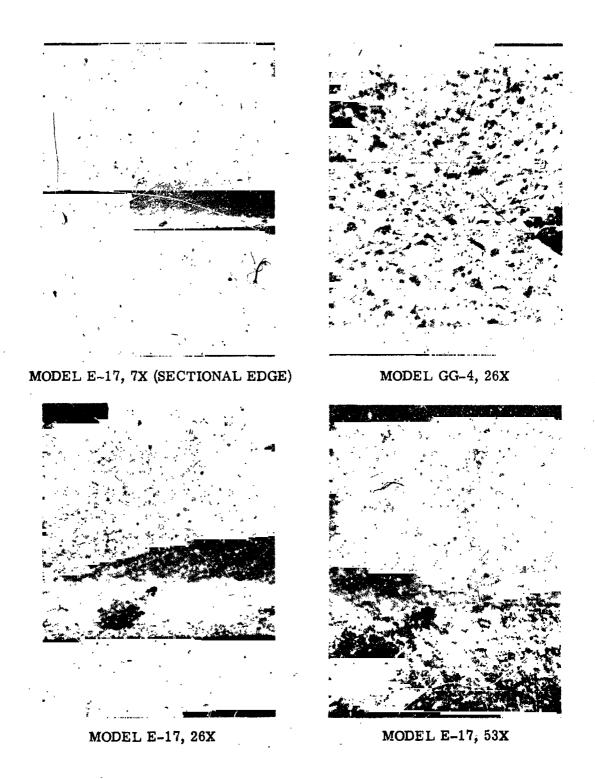


Figure 6-3. Sectional Photomicrographs of Models CG-4 and E-17



VERIFICATION OF LAYERED DEPOSITS OF SALT.

A verification of the spray procedure as the cause of material blistering was made by microscopic examination of sections of several Go - No Go models. The photographs shown in Figure 6-3 are photomicrographs of sectioned edges from the sides of blistered model E-17 and nonblistered model GG-4. Comparison of the photographs shows uniform distribution of the salt in the nonblistered model, which is very clearly distinguishable from the high concentration of salt in the blistered model.

The 26X sectional photograph of model 17-E shows that the spray deposits at the bottom of the specimen contain the predicted concentration of sait of large crystal size. The layers covering this section are seen to be more concentrated in resins. The 53X enlargement of model 17-E clearly distinguishes the levels of material concentrations. These photographs indicate that photomicroscopic examination of applied material presents an effective means to readily distinguish a defective mixing and spraying process.



SECTION VII

QUALITY CONTROL PROGRAM FOR PREPARATION OF "THERMO-LAG" T-500

CHEMICAL AND PHYSICAL ANALYSIS.

The quality control program for the preparation of "THERMO-LAG" prior to the Go-No Go test series consisted of the chemical and physical analyses presented in this section. The chemical analysis of the material with the quality control limits, prior to spray application are:

Viscosity at 77°F Solids (after removal of solvent) NH4BF4 100-180 centipoises 38% minimum 19% minimum.

Following the composition analysis of the material, a test panel of each lot is prepared by spraying and curing, following by the specified chemical and physical tests:

Total solids
Residual solvent

NH4BF4

Differential thermal analysis

Density

Hardness

Tensile strength

X-ray inspection for voids

X-ray inspection for salt coagulation

98% minimum 2% maximum

48-50%

550-600°F at 10°C/MIN

58-63 LB/FT³

500-600 psi

A final check for models fabricated is made for proper dimension and weight.

The Quality Control Inspection sheets for both lot and sample analysis for the blister verification test are shown in Figures C-1 through C-21, Appendix C.

The results of the investigative study on blistered materials shows that an additional step in the quality control of prepared "THERMO-LAG" T-500 is required. Findings of the study shows that photomicrographs of prepared "THERMO-LAG" will provide the required quality control function. The necessary steps to established photomicrographs as a material control measure have been instituted.



APPENDIX A PHOTOGRAPHS OF MODELS TESTED

Figures A-1 through A-19 illustrate the conditions of the models after testing. Models identified with a GG prefix were prepared so as not to blister. Models identified with a D prefix were prepared so as to form large blisters at specific predetermined locations. Models identified with an E prefix were prepared so as to form small blisters at specific predetermined locations.

GG-1A 140 SEC.

Figure A-1. Test Model GG-1A





GG-2 75 SEC.

Figure A-2. Test Model GG-2



GG-4 150 SEC.

Figure A-3. Test Model GG-4



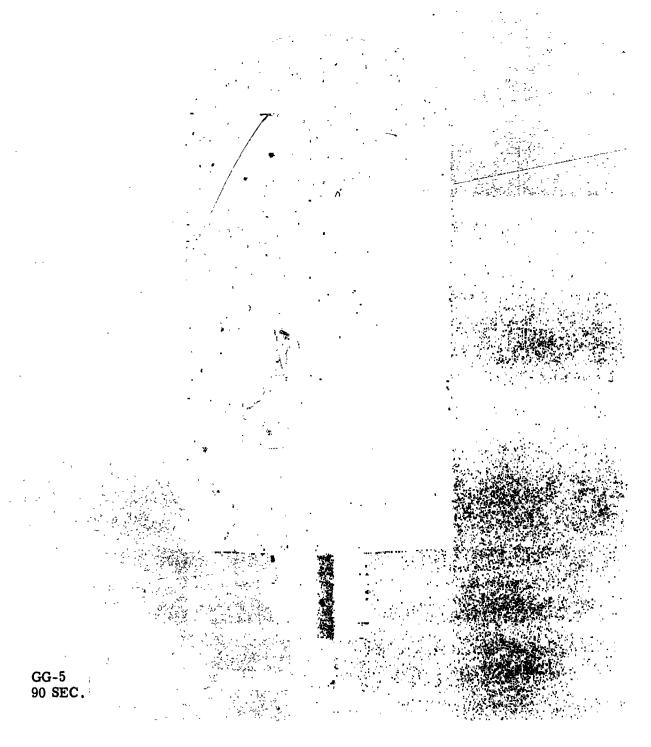
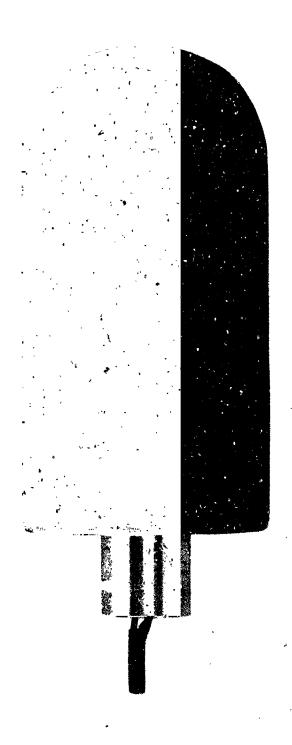


Figure A-4. Test Model GG-5

GG-7 780 SEC.

Figure A-5. Test Model GG-7





GG-8 35 SEC.

Figure A-6. Test Model GG-8

GG-9 ,40 SEC.

Figure A-7. Test Model GG-9



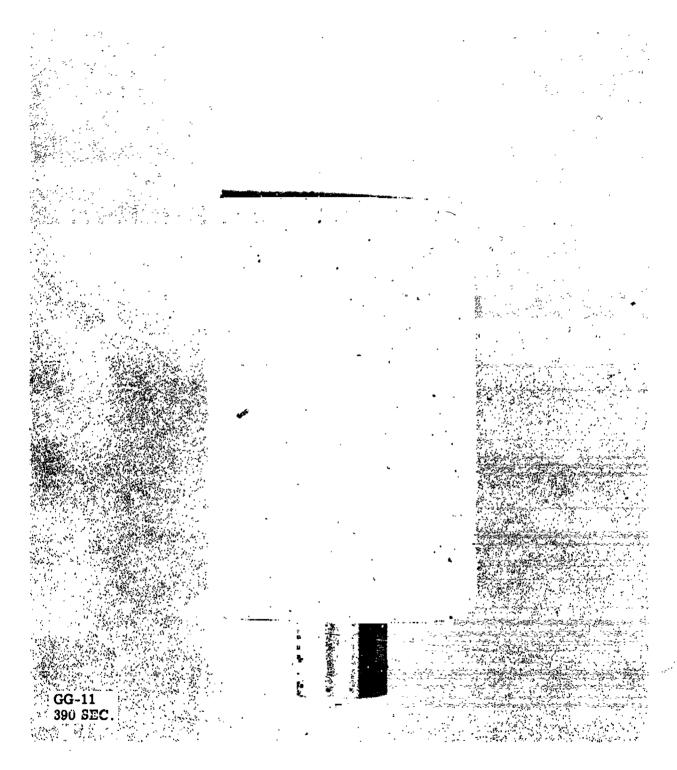


Figure A-8. Test Model GG-11

Figure 4-9. Test Model D-1

1-D 75 SEC.



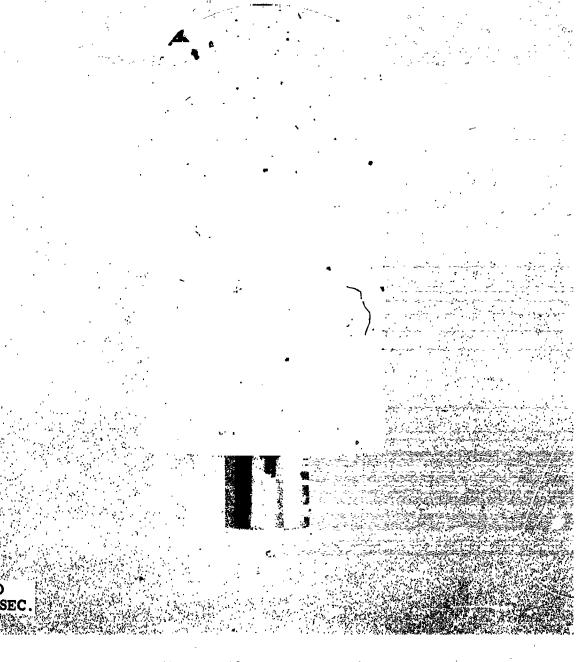


Figure A-10. Test Model D-2

D-2-4 345 SEC.

Figure A-11. Test Model D-2-4





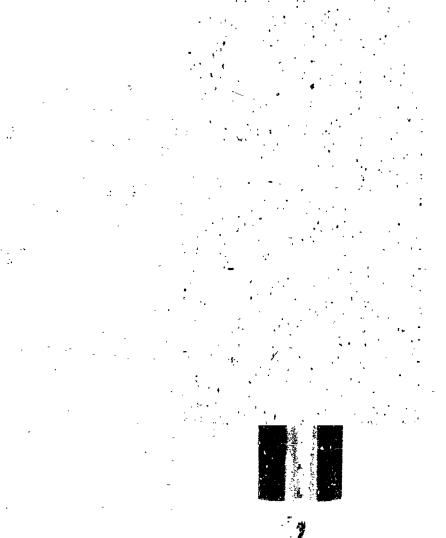
15-E 75 SEC.

Figure A-12. Test Model E-15

16-E 75 SEC.

Figure A-13. Test Model E-16





17-E 50 SEC.

Figure A-14. Test Model E-17



Figure A-15. Test Model E-16

18-E 75 SEC.



9-D 320 SEC.

Figure A-16. Test Model D-9

10-D 385 SEC.

Figure A-17. Test Model D-10





Figure A-18. Test Model D-11

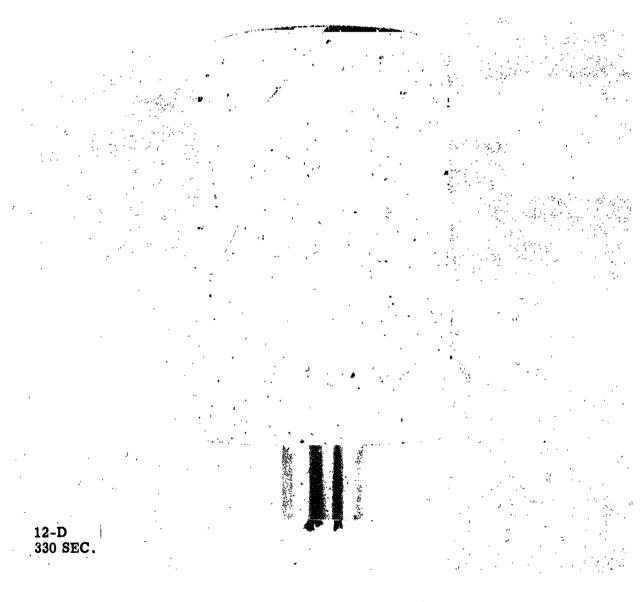


Figure A-19. Test Model D-12



APPENDIX B SUBSTRATE TEMPERATURES OF MODELS TESTED

Graphs of the substrate temperatures of the models tested are shown in Figures B-1 through B-22.

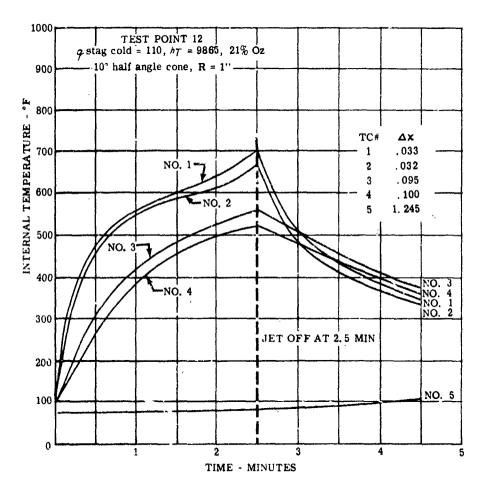


Figure B-1. Sidewall Substrate Temperature - Time History of Model M-9

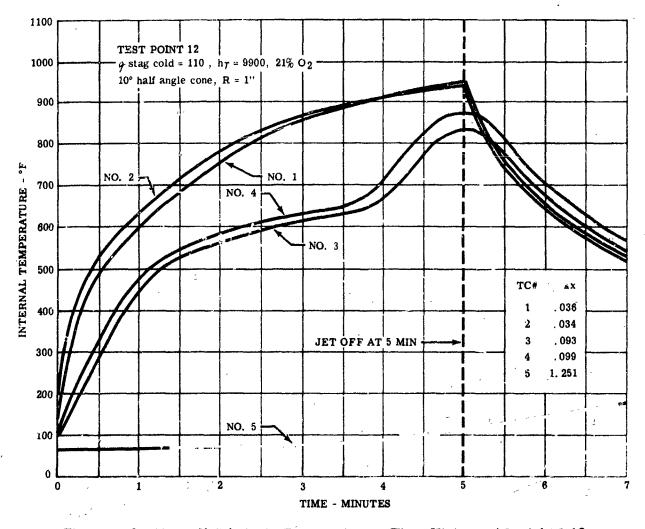


Figure B-2. Sidewall Substrate Temperature - Time History of Model M-10



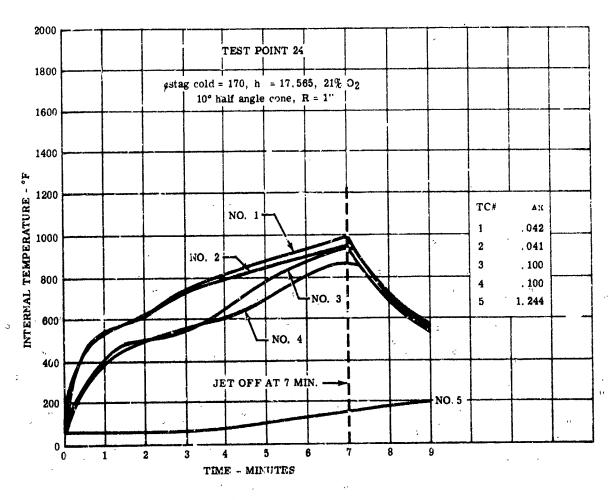


Figure B-3. Sidewall Substra Temperature - Time History of Model M-16

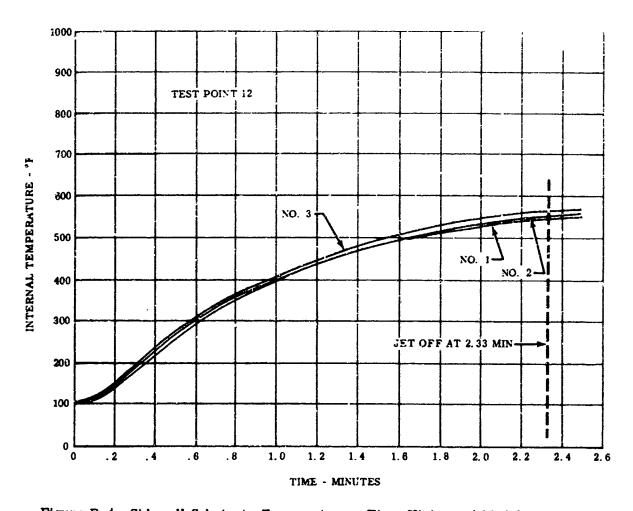


Figure B-4. Sidewall Substrate Temperature - Time History of Model GG-1A



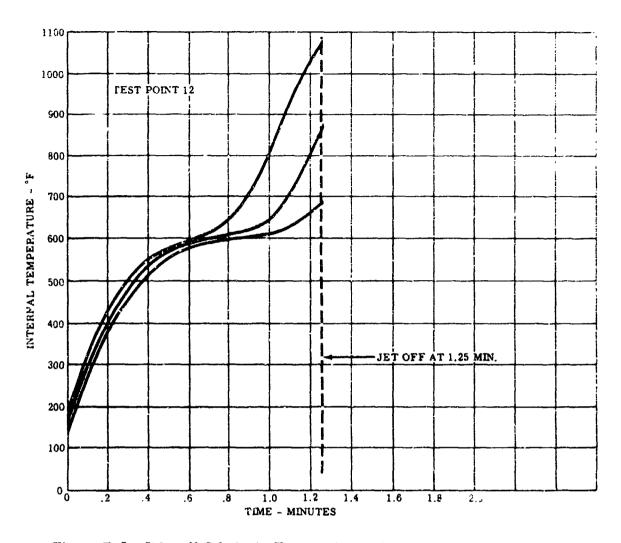


Figure B-5. Sidewall Substrate Temperature - Time History of Model GG-2

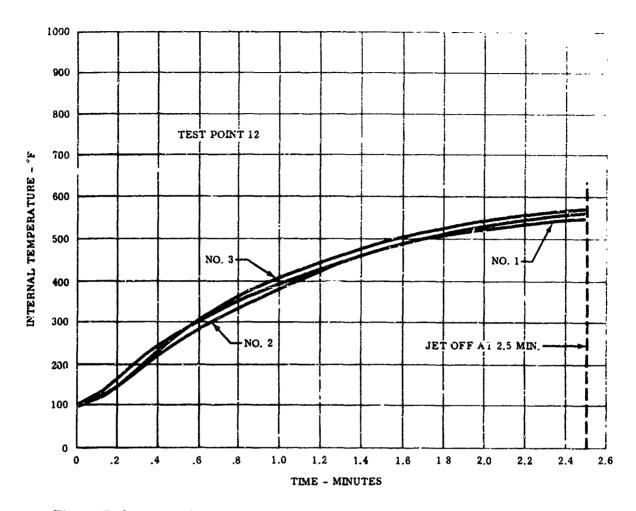


Figure B-6. Sidewall Substrate Temperature - Time History of Model GG-4



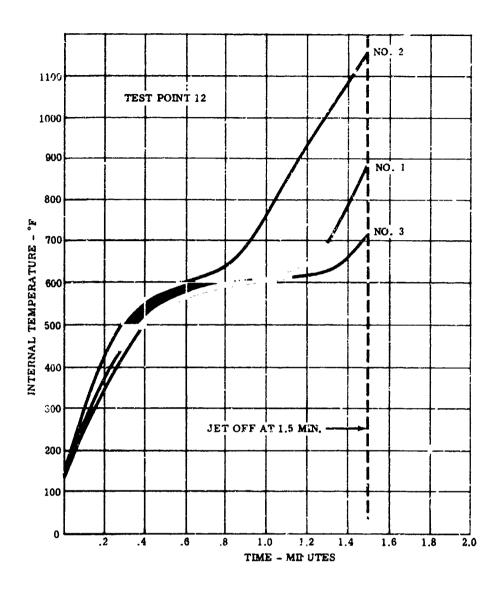


Figure B-7. Sidewall Substrate Temperature - Time History of Model GG-5

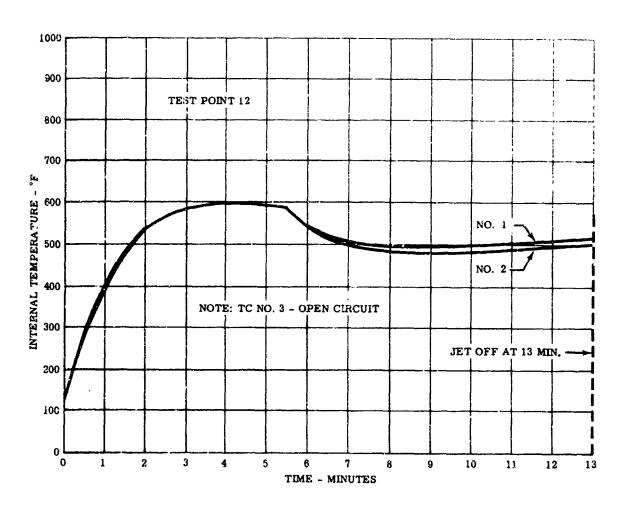


Figure B-8. Sidewall Substrate Temperature - Time History of Model GG-7



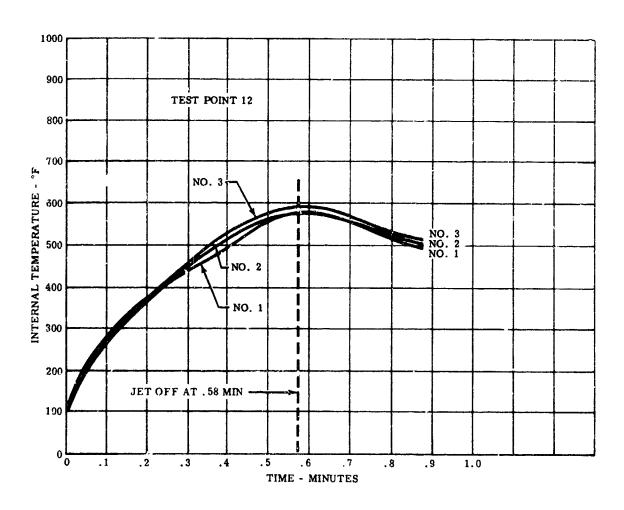


Figure B-9. Sidewall Substrate Temperature - Time History of Model GG-8

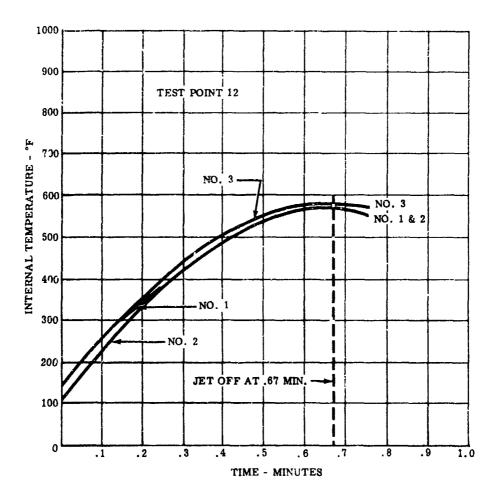
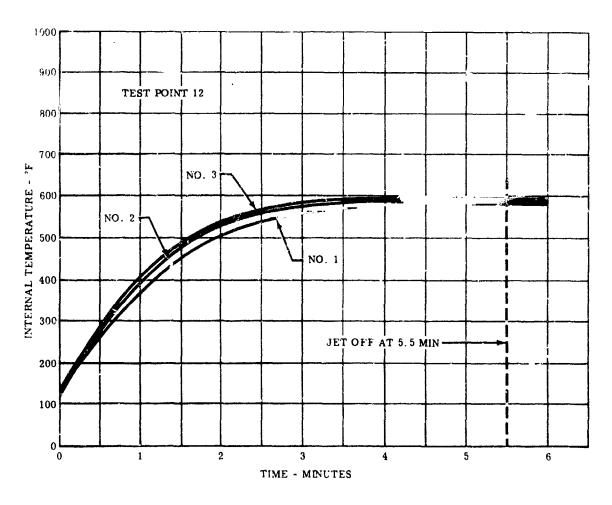


Figure B-10. Sidewall Substrate Temperature - Time History of Model GG-9





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Figure B-11. Sidewall Substrate Temperature - Time History of Model GG-11

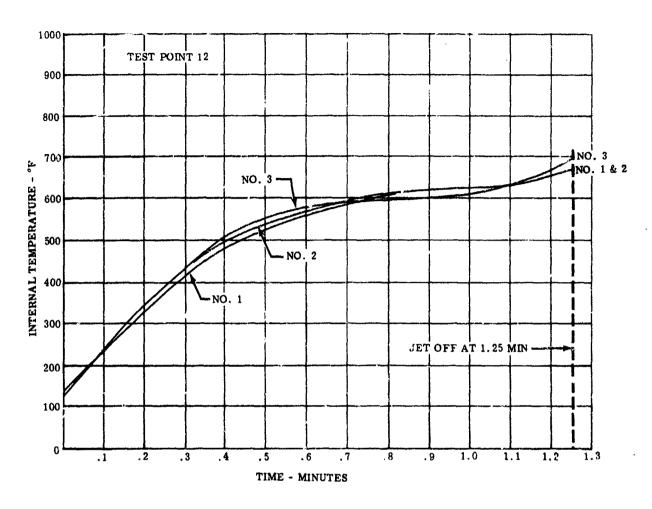


Figure B-12. Sidewall Substrate Temperature - Time History of Model D-1



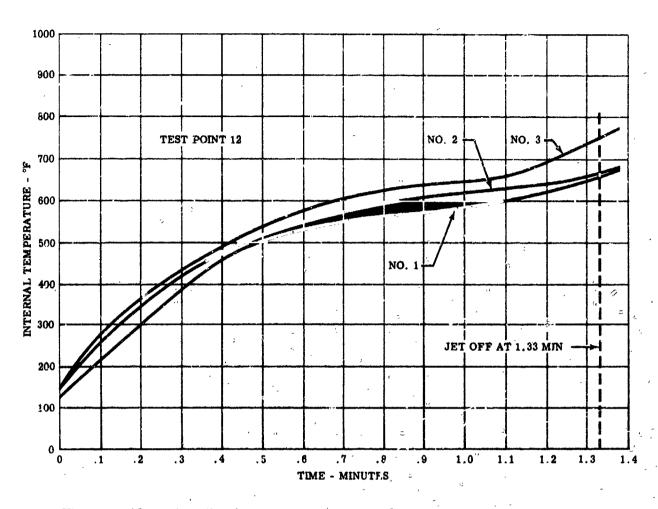


Figure B-13. Sidewall Substrate Temperature - Time History of Model D-2

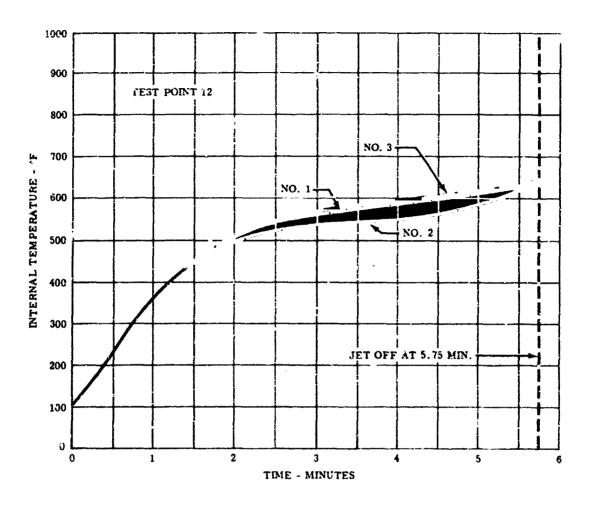


Figure B-14. Sidewall Substrate Temperature - Time History of Model D-2-4



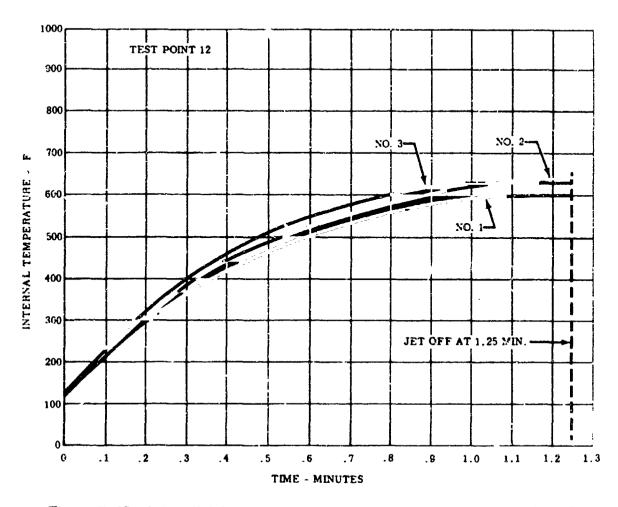


Figure B-15. Sidewall Substrate Temperature - Time History of Model E-15

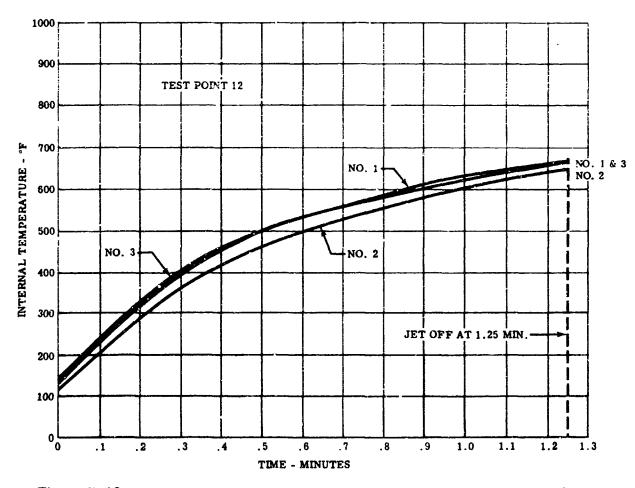


Figure B-16. Sidewall Substrate Temperature - Time History of Model E-16



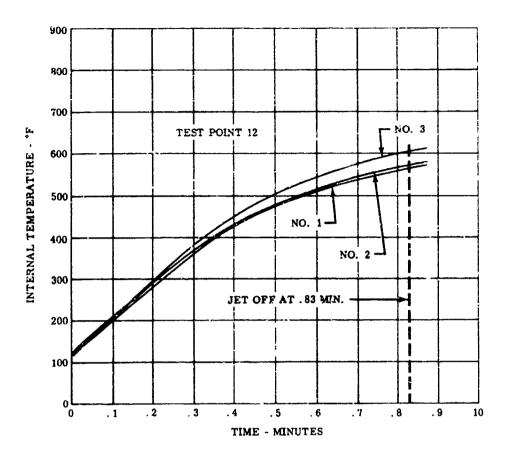


Figure B-17. Sidewall Substrate Temperature - Time History of Model E-17

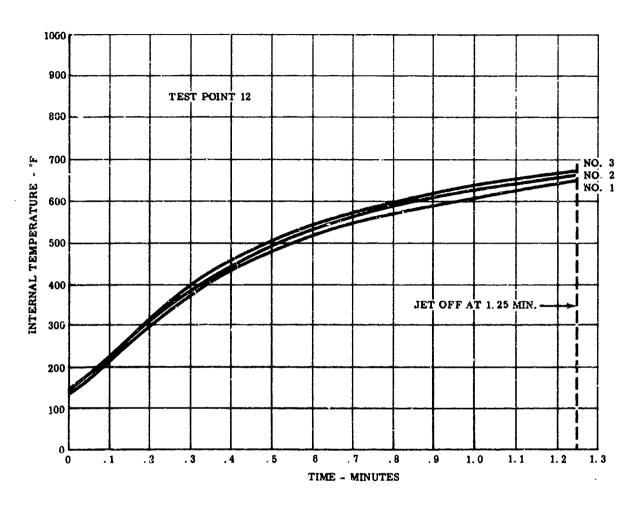


Figure B-18. Sidewall Substrate Temperature - Time History of Model E-18



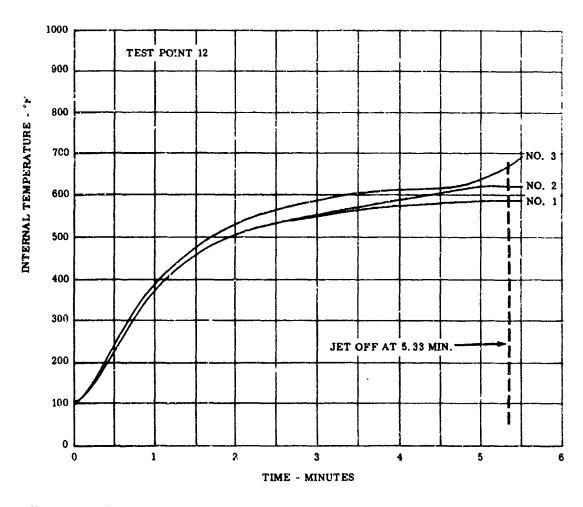


Figure B-19. Sidewall Substrate Temperature - Time History of Model D-9

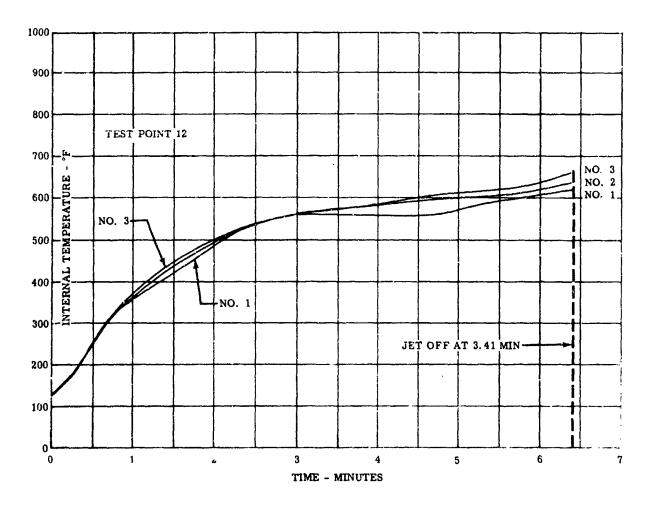


Figure B-20. Sidewall Substrate Temperature - Time History of Model D-10



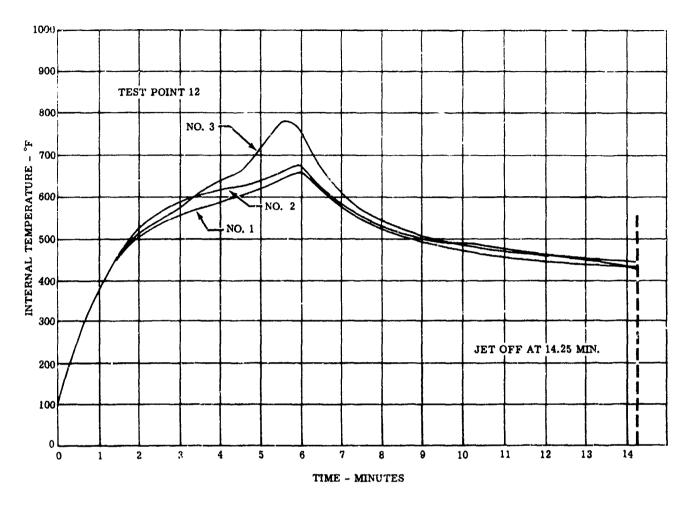


Figure B-21. Sidewall Substrate Temperature - Time History of Model D-11

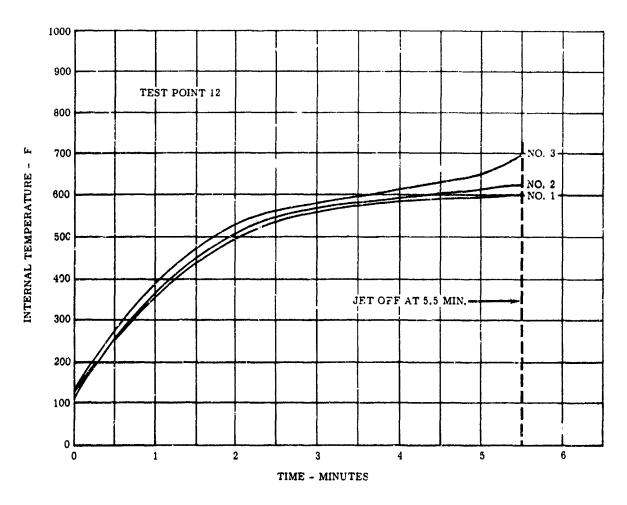


Figure B-22. Sidewall Substrate Temperature - Time History of Model D-12



APPENDIX C QUALITY CONTROL SHEETS

The Quality Control Sheets for the models tested are presented in Figures C-1 through C-21.

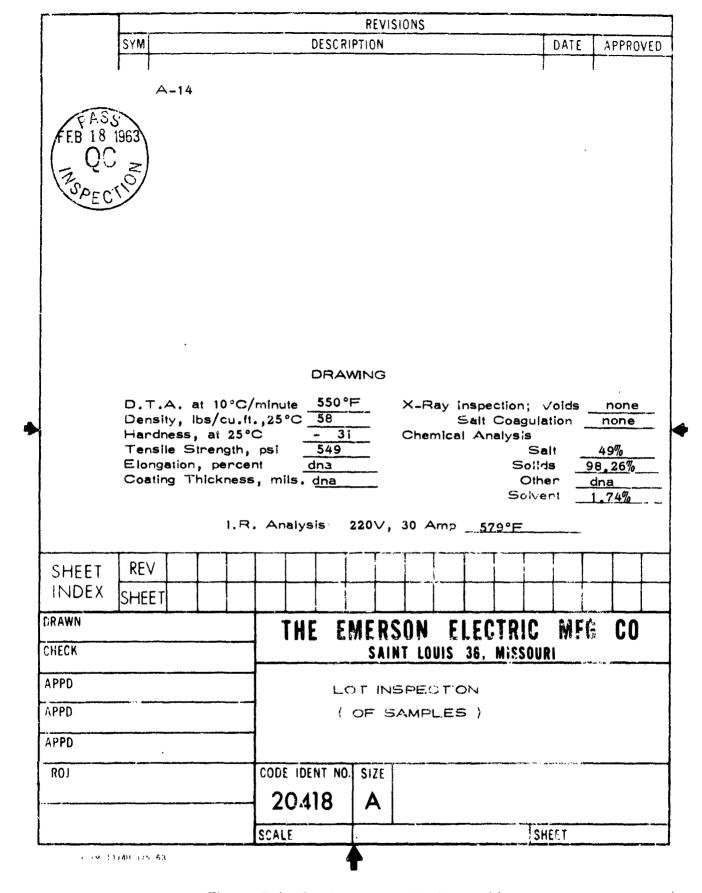


Figure C-1. Lot Inspection Number A-14

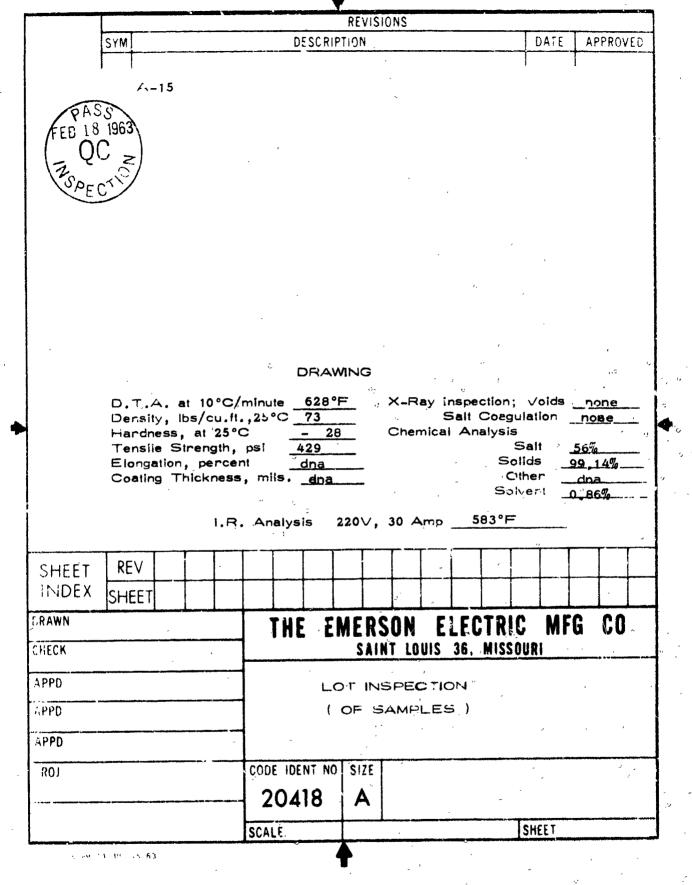


Figure C-2. Lot Inspection Number A-15

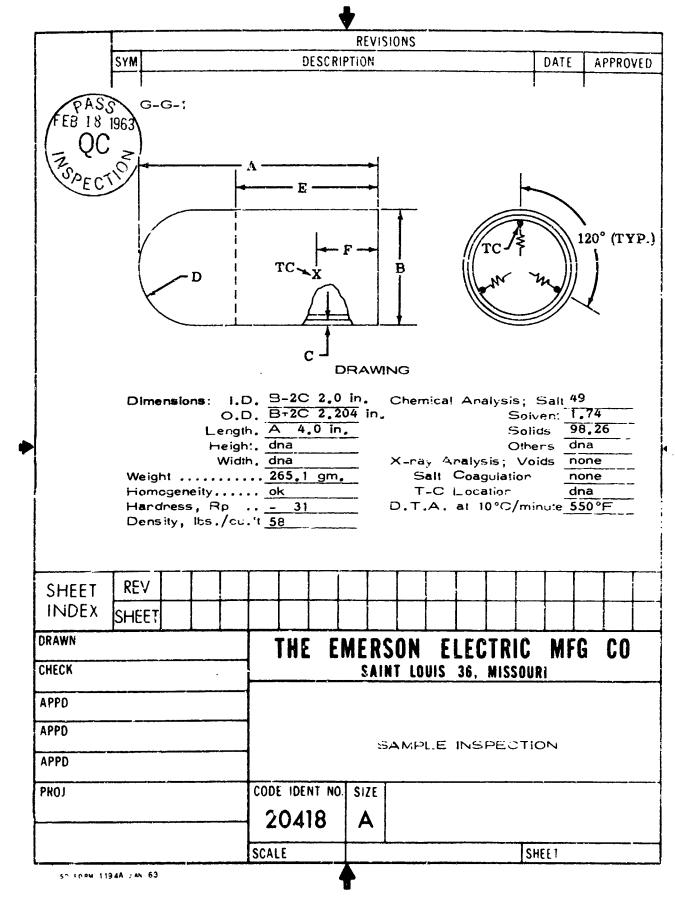


Figure C-3. Sample Inspection for Model G-G-1

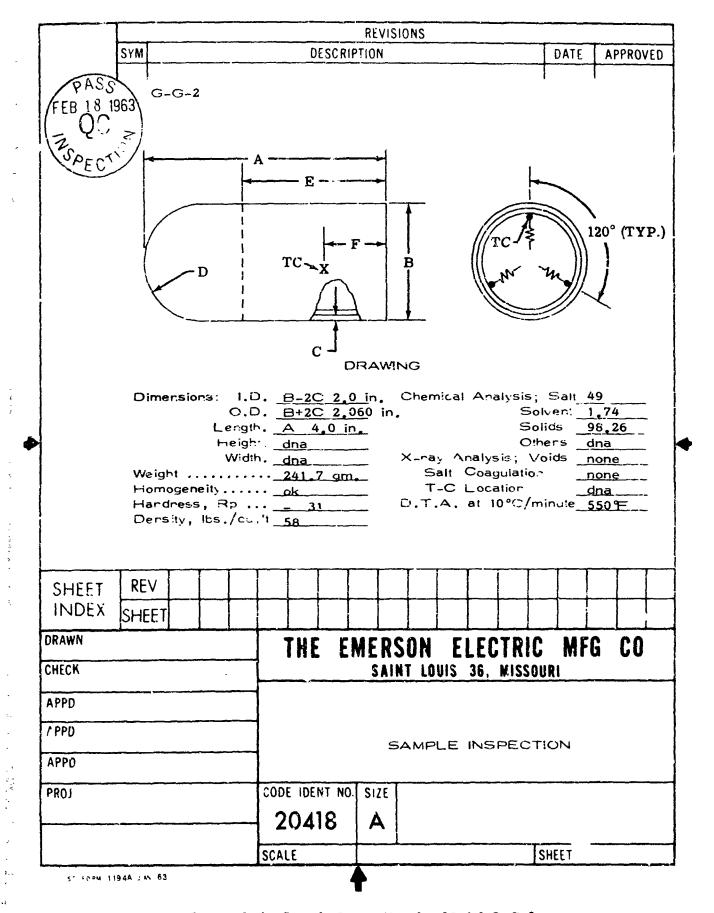


Figure C-4. Sample Inspection for Model G-G-2

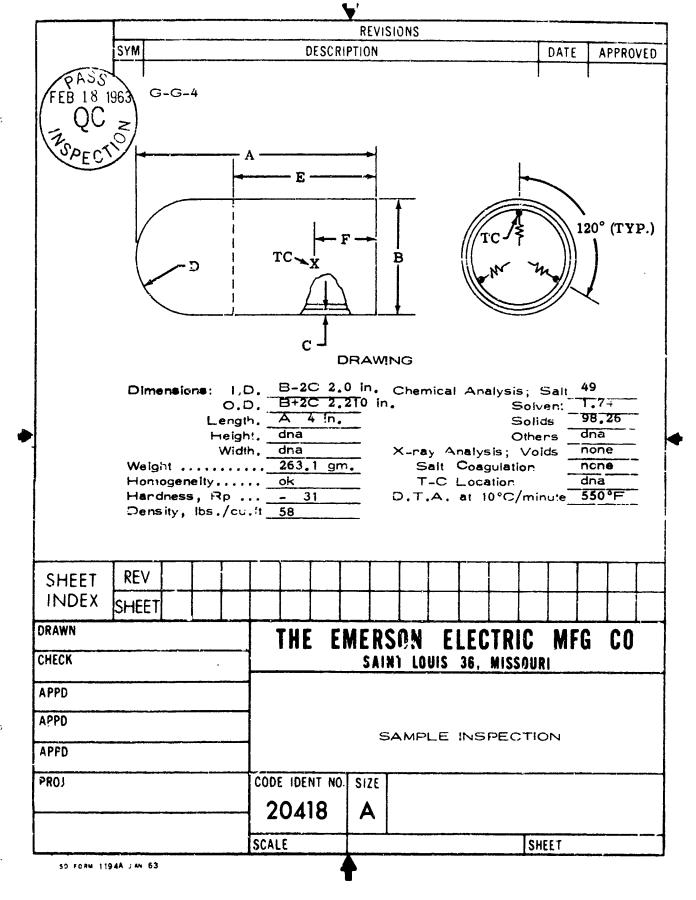


Figure C-5. Sample Inspection for Model G-G-4

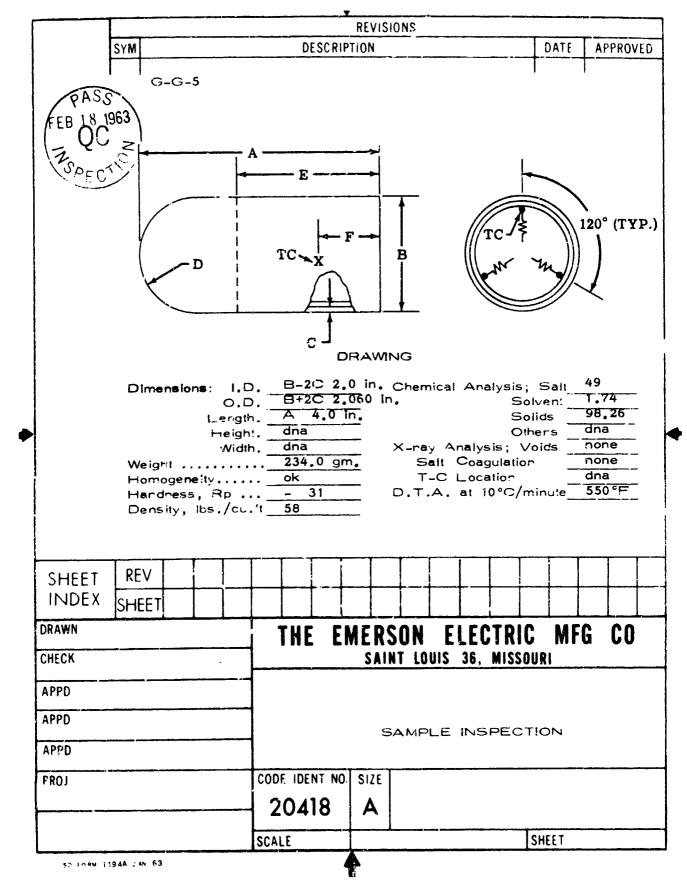


Figure C-6. Sample Inspection for Model G-G-5

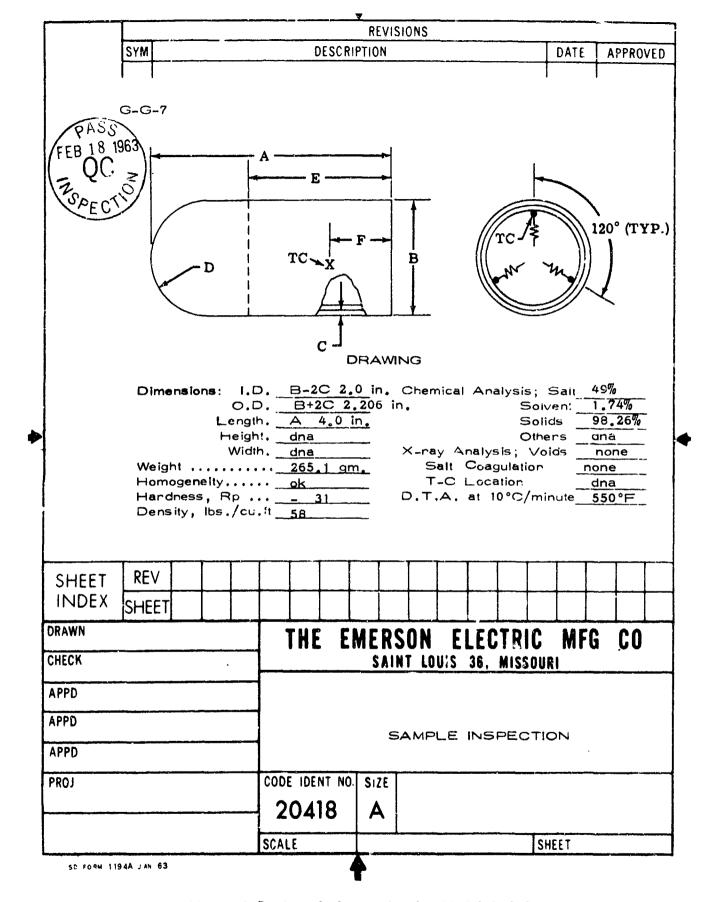


Figure C-7. Sample Inspection for Model G-G-7

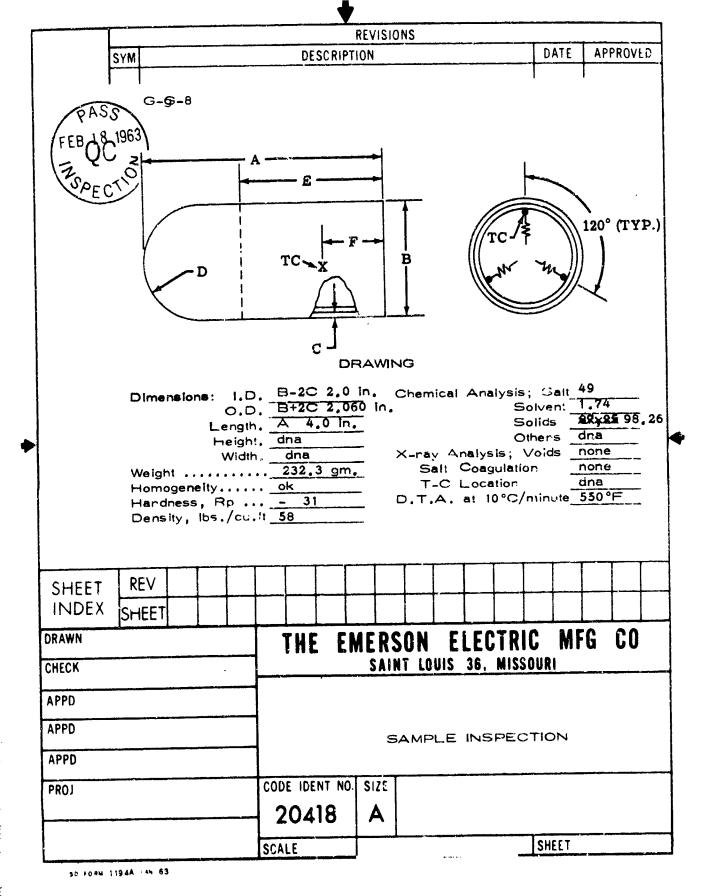


Figure C-8. Sample Inspection for Model G-G-8

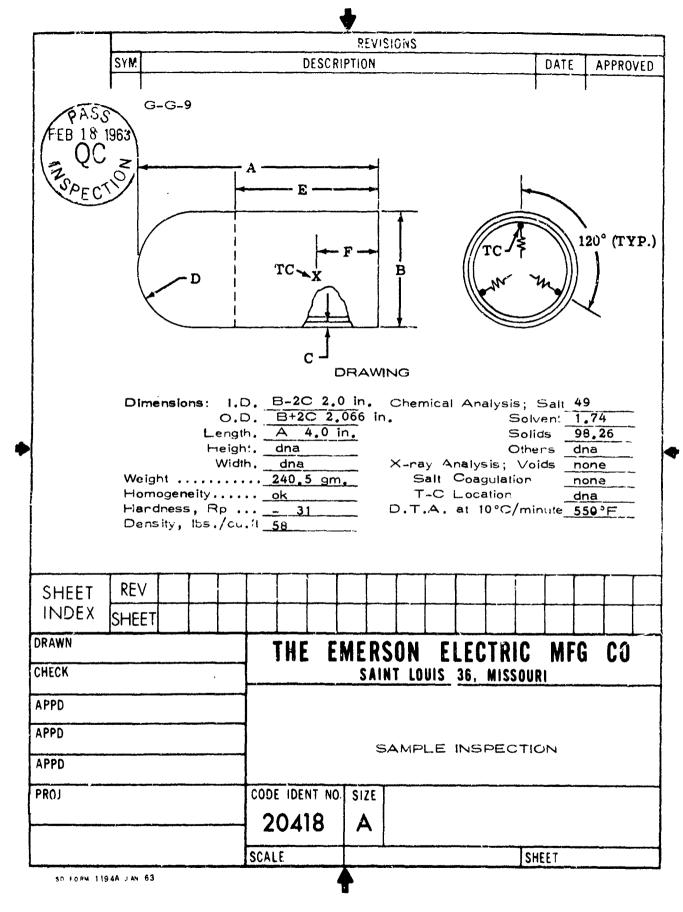


Figure C-9. Sample Inspection for Model G-G-9

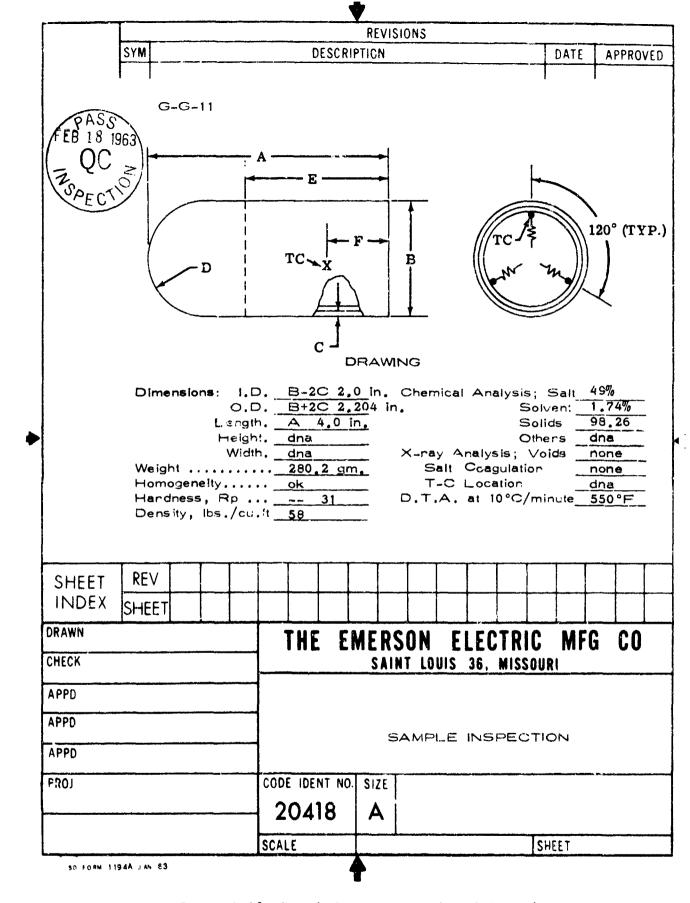


Figure C-10. Sample Inspection for Model G-G-10

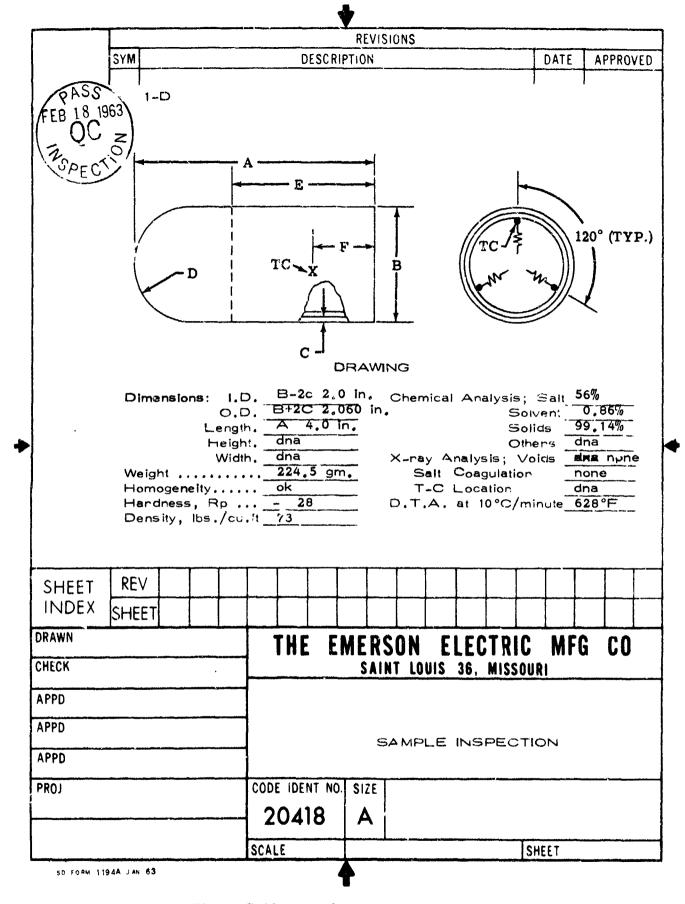


Figure C-11. Sample Inspection for Model 1-D

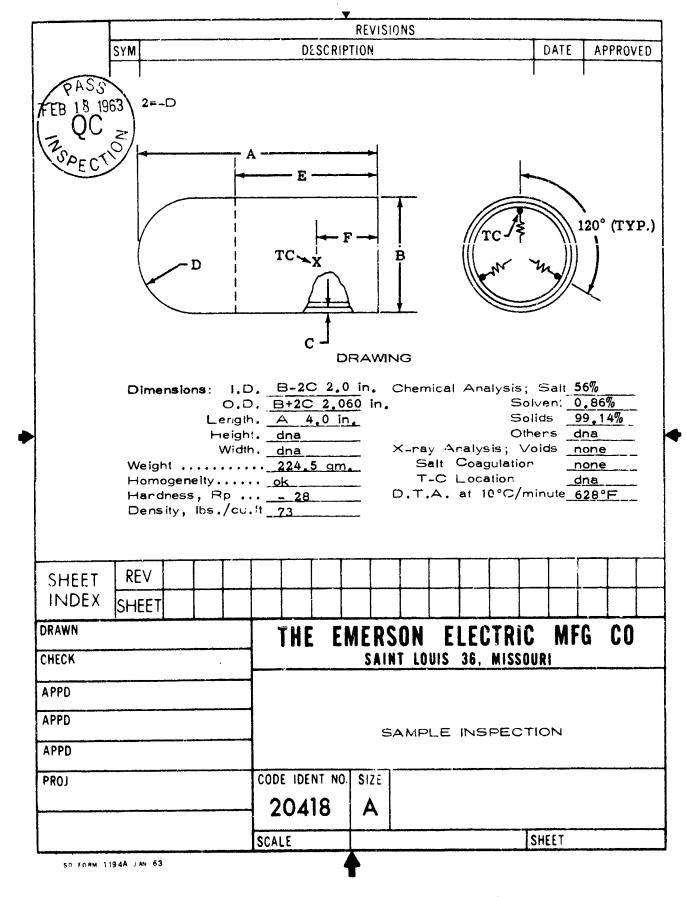


Figure C-12. Sample Inspection for Model 2-D

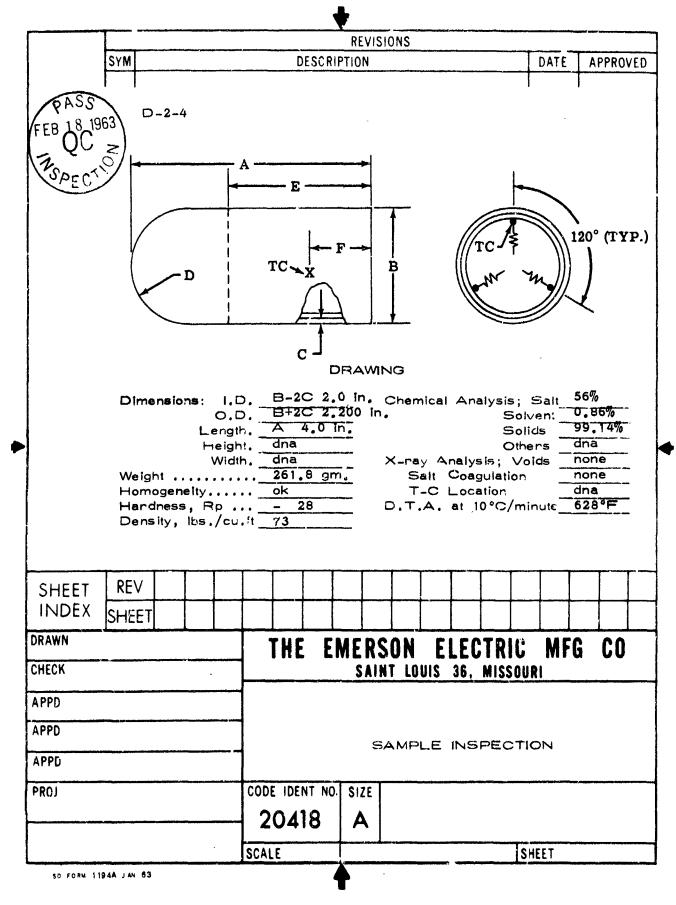


Figure C-13. Sample Inspection for Model D-2-4

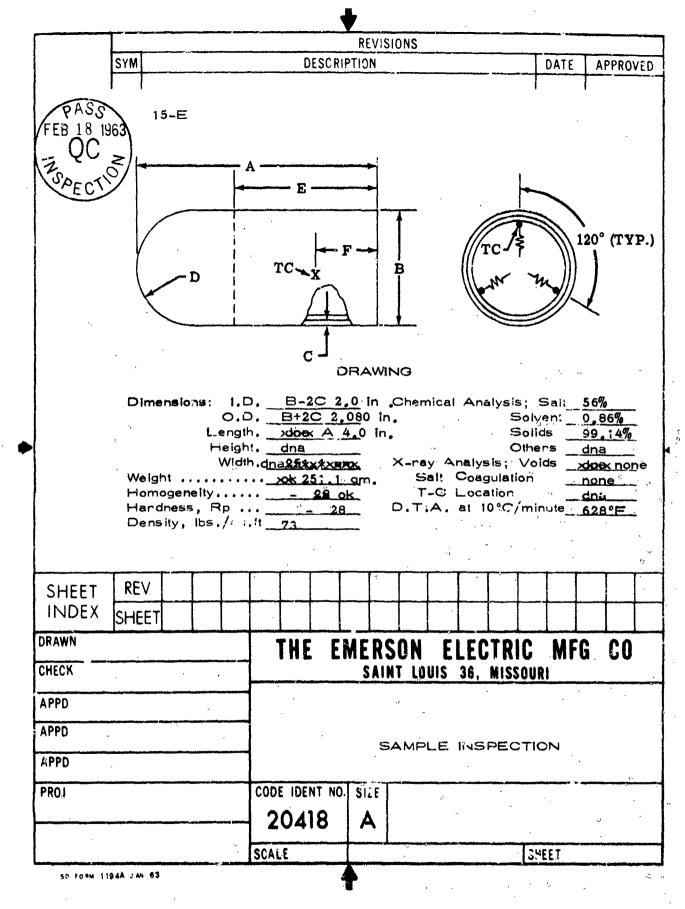


Figure C-14. Sample Inspection for Model 15-E

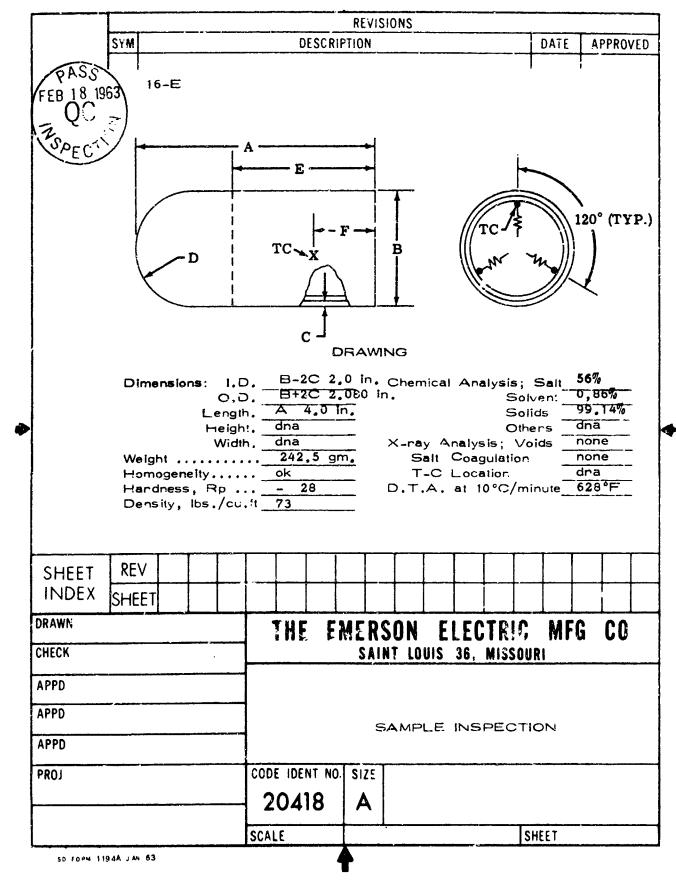


Figure C-15. Sample Inspection for Model 16-E

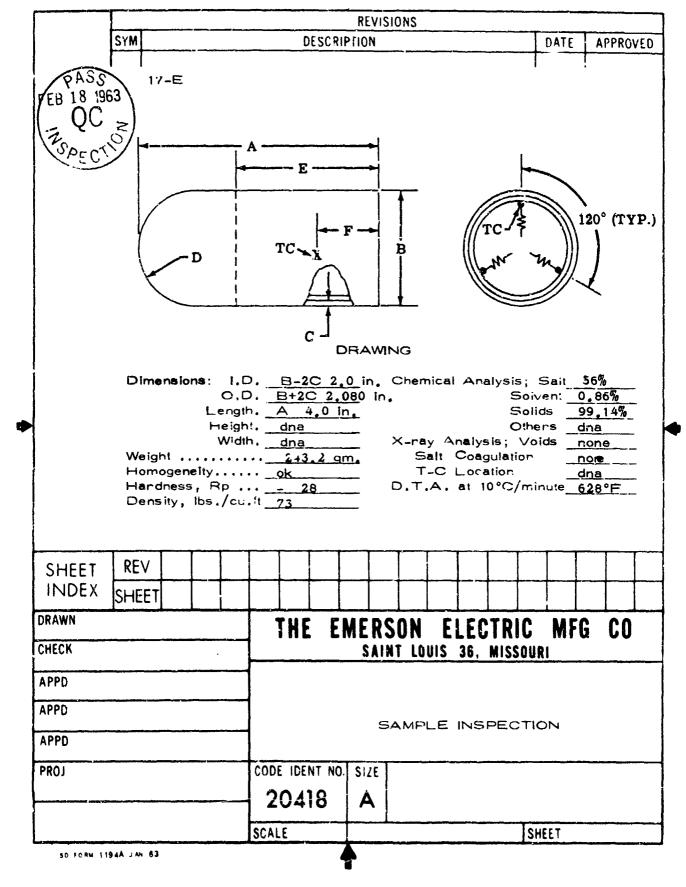


Figure C-16. Sample Inspection for Model 17-E

医病性的复数 衛門工作者 上級軍人人科公公 五五十

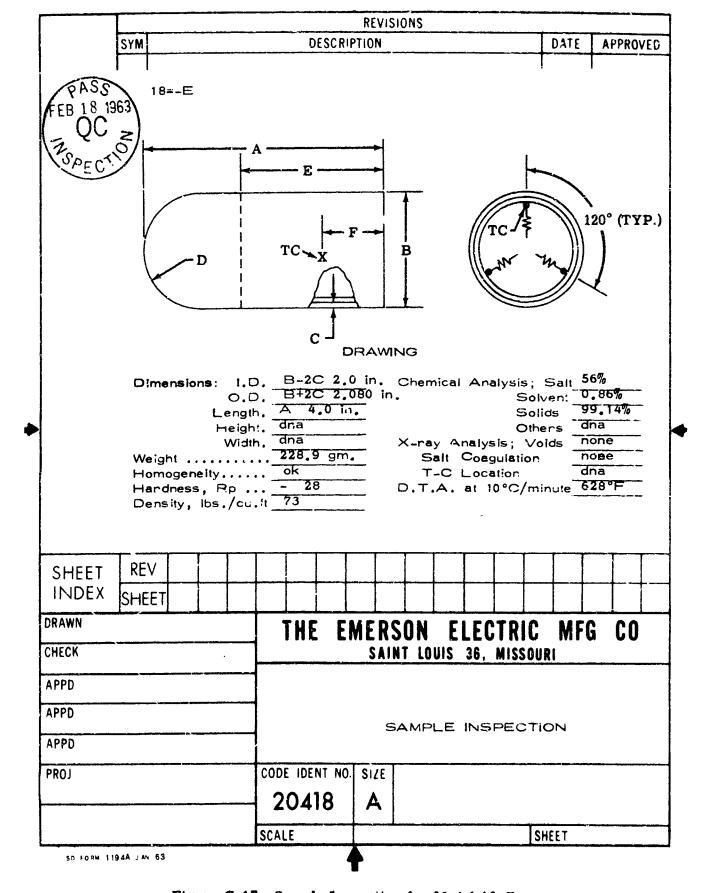


Figure C-17. Sample Inspection for Model 18-E

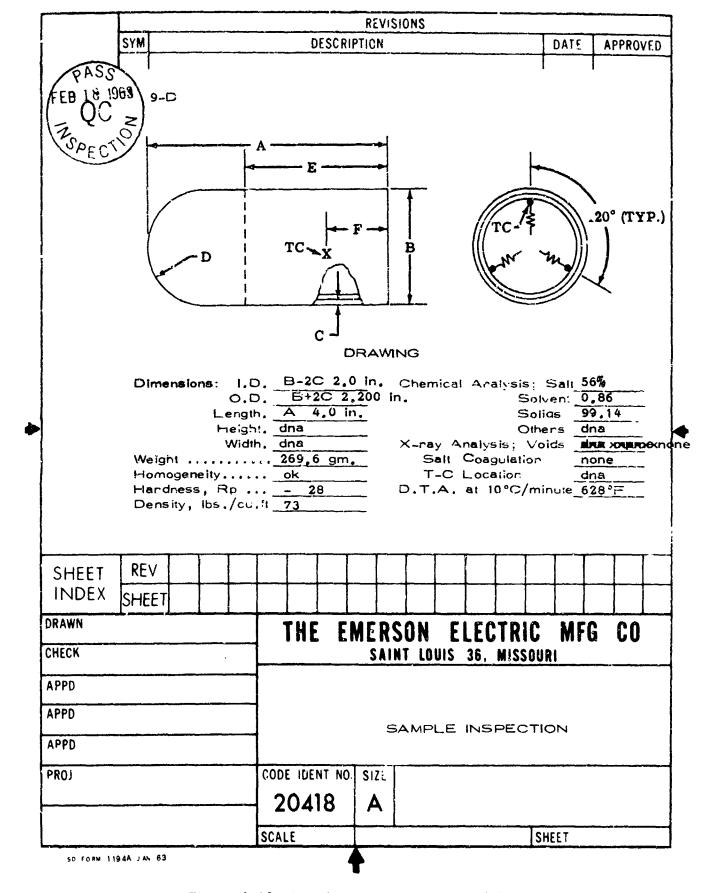


Figure C-18. Sample Inspection for Model 9-D

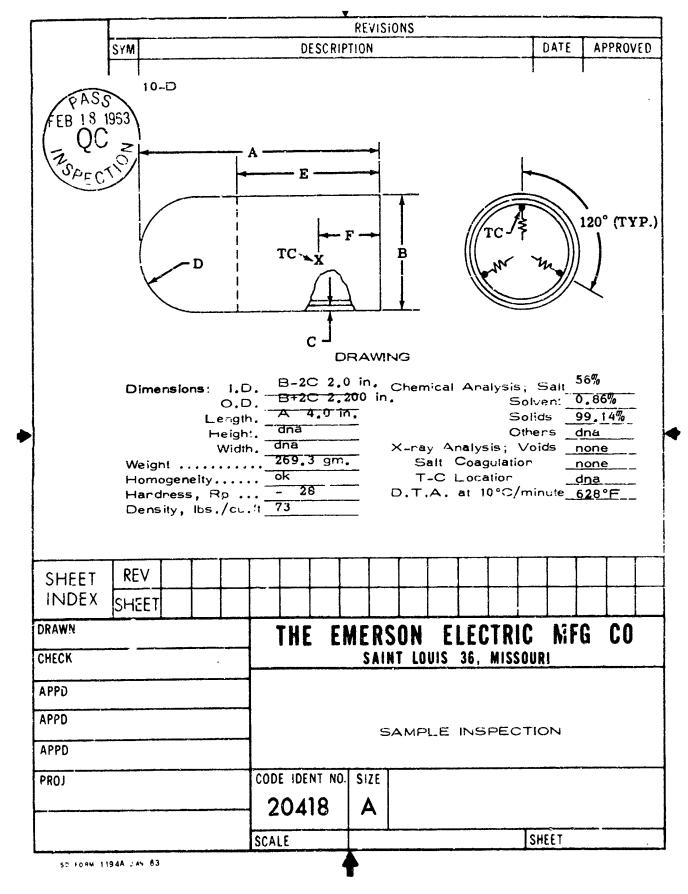


Figure C-19. Sample Inspection for Model 10-D

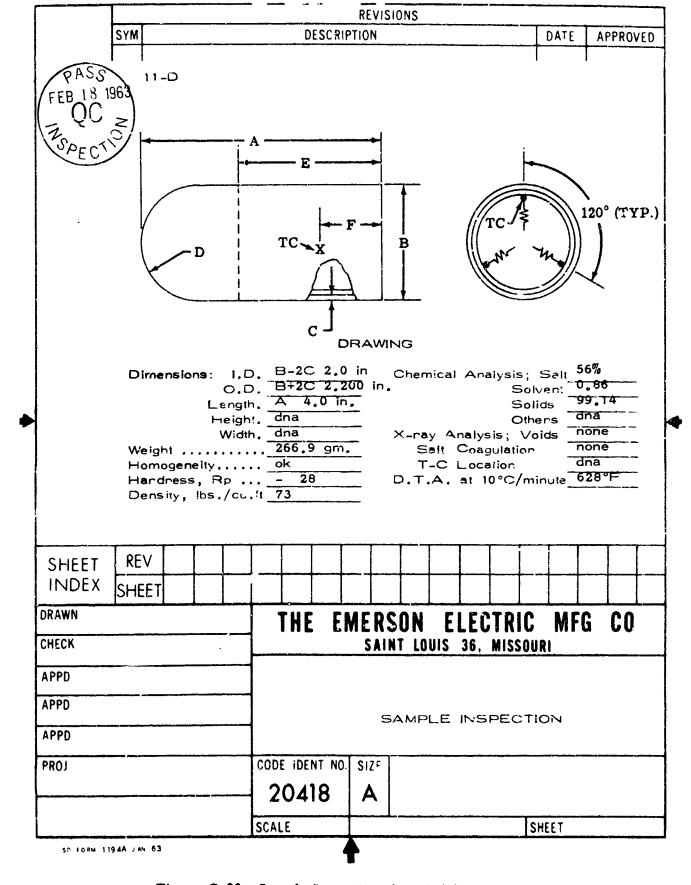


Figure C-20. Sample Inspection for Model 11-D

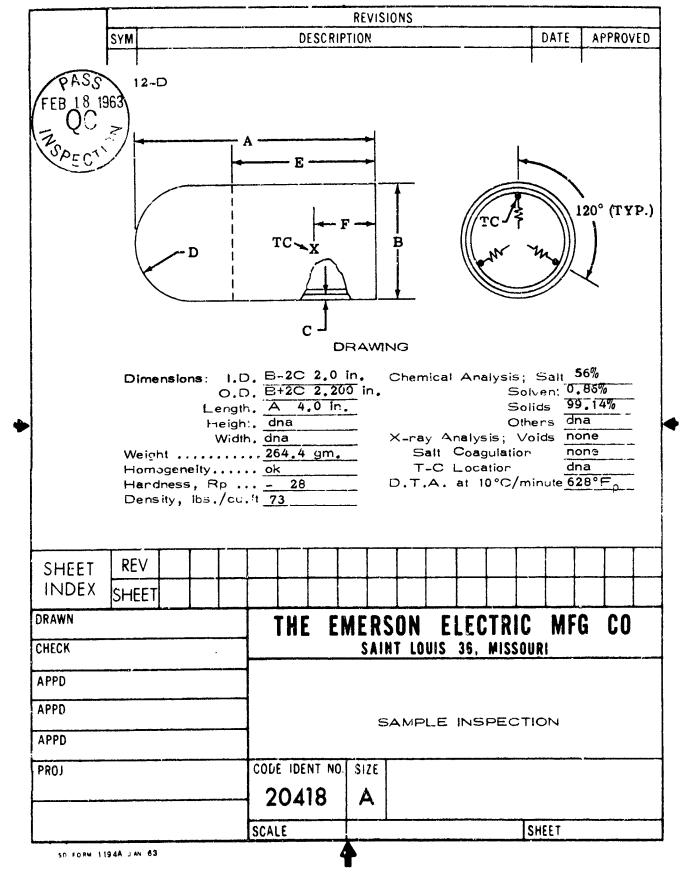


Figure C-21. Sample Inspection for Model 12-D